



February 5, 2008

**PHASE II
SAMPLING AND ANALYSIS PLAN
FOR OPERABLE UNIT 3
LIBBY ASBESTOS SUPERFUND SITE**

Part A: Surface Water and Sediment

**Prepared by
U.S. Environmental Protection Agency
Region 8
Denver, CO**



With Technical Assistance from:

**Syracuse Research Corporation
Denver, CO**



and

**NewFields Boulder LLC
Boulder, CO**



APPROVAL PAGE

Part A of the Phase II Sampling and Analysis Plan for Operable Unit 3 of the Libby Asbestos Superfund Site has been prepared by the U.S. Environmental Protection Agency, Region 8, with technical support from Syracuse Research Corporation and NewFields Boulder LLC, and is approved without conditions.

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LIST OF ATTACHMENTS

LIST OF ACRONYMS

BTEX	Benzene, toluene, ethyl benzene, and xylenes
CAR	Corrective Action Request
CCV	Continuing Calibration Verification
CSF	Close Support Facility
cfs	cubic feet per second
COC	Chain of Custody
CSM	Conceptual Site Model
DO	Dissolved Oxygen
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDXA	Energy Dispersive X-Ray Analysis
EMAP	Environmental Monitoring and Assessment Program
EPA	U.S. Environmental Protection Agency
EPH	Extractable Petroleum Hydrocarbons
USFWS	U.S. Fish and Wildlife Service
FS	Feasibility Study
FSDS	Field Sample Data Sheets
FSP	Field Sampling Plan
FTP	File Transfer Protocol
GC/MS	Gas chromatography/mass spectroscopy
GO	Grid opening
HASP	Health and Safety Plan
ICV	Initial Calibration Verification
ID	Identification
IL	Inter-laboratory
ISO	International Organization for Standardization
IS	Internal Standard
LA	Libby Amphibole
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MCL	Maximum Contaminant Level
MDEQ	Montana Department of Environmental Quality
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MTBE	Methyl tert-butyl ether
NVLAP	National Voluntary Laboratory Accreditation Program
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PDF	Portable Document Format
PE	Performance Evaluation
PLM	Polarized Light Microscopy

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PLM-VE	Polarized Light Microscopy Visual Area Estimation Method
PPE	Personal Protective Equipment
PR	Percent Recovery
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QATS	Quality Assurance Technical Support
QC	Quality Control
RD	Recount Different
RF	Response Factors
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RS	Recount Same
RSD	Relative Standard Deviation
SAED	Selective Area Electron Diffraction
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SPP	Soil Preparation Plan
TAL	Target Analyte List
TCL	Target Compound List
TEH	Total Extractable Hydrocarbons
TEM	Transmission Electron Microscopy
TWF	Time-Weighting Factor
USGS	U.S. Geological Survey
VOC	Volatile Organic Compound
VPH	Volatile Petroleum Hydrocarbons

**PHASE II
SAMPLING AND ANALYSIS PLAN
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PART A: SURFACE WATER AND SEDIMENT

1.0 PROJECT OVERVIEW

1.1 Purpose of this Document

This document is Part A of the Phase II Sampling and Analysis Plan (SAP) for the collection and analysis of samples of environmental media within Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site near Libby, Montana. The purpose of Part A of the Phase II SAP for OU3 is to guide the collection of data on mining-related contaminants in surface water and sediment in streams and ponds that are impacted by releases from the mined area. Data on other environmental media of potential concern in OU3 will be collected as detailed in Part B of the Phase II SAP for OU3. These data will be used to support a remedial investigation of OU3, the goal of which is to characterize the nature and extent of mining-related contamination in OU3, and to characterize the nature and level of risk posed by mining-related contamination to human and ecological receptors in OU3.

This SAP contains the elements required for both a field sampling plan (FSP) and quality assurance project plan (QAPP). This SAP has been developed in accordance with the EPA Requirements for Quality Assurance Project Plans (EPA 2001) and the Guidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4 (EPA 2006). The SAP is organized as follows:

- Section 1 – Project Overview
- Section 2 – Background and Problem Definition
- Section 3 – Summary of Phase I Data
- Section 4 – Data Quality Objectives
- Section 5 – Sampling Program
- Section 6 – Laboratory Analysis Requirements
- Section 7 – Site-Specific Toxicity Testing requirements
- Section 8 – Quality Control
- Section 9 – Data Management
- Section 10 – Assessment and Oversight
- Section 11 – Data Validation and Usability
- Section 12 – References

1.2 Project Management and Organization

Project Management

EPA is the lead regulatory agency for Superfund activities within OU3. The EPA Remedial Project Manager (RPM) for OU3 is Bonita Lavelle, EPA Region 8. Ms. Lavelle is a principal data user and decision-maker for Superfund activities within OU3.

The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities within OU3. The MDEQ Project Manager for OU3 is Catherine LeCours. EPA will consult with MDEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities within OU3.

EPA has entered into an Administrative Order on Consent (AOC) with Respondents W.R. Grace & Co.-Conn. and Kootenai Development Corporation (KDC). Under the terms of the AOC, W.R. Grace & Co.-Conn. and KDC will implement this SAP. The designated Project Coordinator for Respondents W.R. Grace & Co.-Conn. and KDC is Robert Marriam of Remedium Group, Inc.

Technical Support

EPA will be supported in this project by a number of contractors, including:

- Syracuse Research Corporation (SRC) will assist in the development of sampling and analysis plans, and in the evaluation and interpretation of the data.
- NewFields Boulder LLC, working as a subcontractor to SRC, will provide support in sampling and analysis, mapping and other GIS applications, and design and evaluation of the feasibility study.
- Department of Transportation, John A. Volpe National Transportation Systems Center (Volpe) will provide management and coordination of resources for field oversight of sampling activities.
- Camp Dresser & McKee, Inc. (CDM) will provide on-site support and oversight for field sampling activities.

Field Sampling Activities

All field sampling activities described in this SAP will be performed by W.R. Grace & Co.-Conn. and KDC, in strict accord with the sampling plans developed by EPA. W.R. Grace & Co.-Conn. and KDC will be supported in this field work by MWH Global, Inc. (MWH). Individuals responsible for implementation of field sampling activities are listed below:

- MWH Field Supervisor: John D. Garr
- MWH Field Quality Control Officer: Jeremy S. Collyard
- MWH Quality Assurance Officer: Stephanie A. Boehnke

EPA Field Oversight Contractor

The on-site point of contact for access to the mine and the coordinator of field oversight activities for OU3 Phase I sampling is Courtney Zamora of Volpe. Oversight of field sampling activities will be provided by CDM staff, under the supervision of Nick Raines.

Sample Preparation and Analysis

All samples collected as part of the Phase II investigation will be sent for preparation and/or analysis at laboratories selected and approved by EPA.

- All analyses of samples for asbestos will be performed by EMSL Analytical, Inc.
- All analyses of samples for non-asbestos analytes will be performed by Energy Laboratories, Inc. (ELI)
- All samples of soil or soil-like media to be analyzed for non-volatile analytes will be prepared for analysis by EPA's soil preparation facility in Denver, CO, operated by CDM.
- All validation and verification activities for asbestos and non-asbestos data will be performed by SRC or their subcontractors.

Data Management

Administration of the master OU3 database for OU3 will be performed by EPA contractors (SRC and NewFields). The primary database administrator will be Lynn Woodbury. She will be responsible for sample tracking, uploading new data, performing error checks to identify inconsistent or missing data, and ensuring that all questionable data are checked and corrected as needed.. When the OU3 database has been populated, checked and validated, relevant asbestos data will be transferred into the Libby2 database for final storage.

2.0 BACKGROUND AND PROBLEM DEFINITION

2.1 Site Description

Libby is a community in northwestern Montana that is located near a large open-pit vermiculite mine. Vermiculite from the mine at Libby is known to be contaminated with amphibole asbestos that includes several different mineralogical classifications, including richterite, winchite, actinolite and tremolite. For the purposes of EPA investigations at the Libby Superfund Site, this mixture is referred to as Libby Amphibole (LA).

Historic mining, milling, and processing of vermiculite at the site are known to have caused releases of vermiculite and LA to the environment. Inhalation of LA associated with the vermiculite is known to have caused a range of adverse health effects in exposed humans, including workers at the mine and processing facilities (Amandus and Wheeler 1987, McDonald et al. 1986, McDonald et al. 2004, Sullivan 2007, Rohs et al. 2007), as well as residents of Libby (Peipens et al. 2003). Based on these adverse effects, EPA listed the Libby Asbestos Site on the National Priorities List in October 2002.

Starting in 2000, EPA began taking a range of cleanup actions at the site to eliminate sources of LA exposure to area residents and workers using CERCLA (or Superfund) authority. For operational convenience, the site has been divided into a number of Operable Units (OUs). In the early stages, efforts were focused mainly on wastes remaining at former vermiculite processing areas including OU1 (the export plant) and OU2 (the screening plant). As work progressed, attention soon shifted to cleanup of current homes and workplaces in the main residential/commercial areas of Libby (OU4).

OU3 includes the former open pit vermiculite mine that is located northeast of the community of Libby, as well as nearby lands that have been impacted by releases and subsequent migration of hazardous substances and/or pollutants or contaminants from the mine, including ponds, Rainy Creek, Carney Creek, Fleetwood Creek, and the Kootenai River. Rainy Creek Road is also included in OU3. Figure 2-1 shows the location of the mine and an initial boundary for OU3. This boundary is preliminary, and may be revised as data are obtained on the nature and extent of mining-related released of chemical contaminants from the mined area into the environment. The final geographic boundary of OU3 has not yet been defined but will be based primarily upon the extent of contamination associated with releases from the former vermiculite mine as determined in the remedial investigation (RI) of OU3.

2.2 Basis for Concern at OU3

EPA is concerned with environmental contamination in OU3 because the area is used by humans for logging and a variety of recreational activities, and also because the area is suitable habitat

for a wide range of ecological receptors (both aquatic and terrestrial). Contaminants of potential concern to EPA in OU3 include not only LA, but any other mining-related contaminants that may have been released to the environment.

2.3 Scope and Strategy of the RI at OU3

As noted above, EPA is conducting an RI in OU3 in order to characterize the nature and extent of environmental contamination in OU3 and to evaluate risks to humans and ecological receptors from mining-related contaminants in the environment.

The first round of RI sampling (referred to as Phase I) in OU3 was performed in the fall of 2007. This round of sampling was performed in accord with the Phase I Sampling and Analysis Plan for Operable Unit 3 (USEPA 2007). The primary goal of the Phase I investigation was to obtain preliminary data on the levels and spatial distribution of asbestos and also other non-asbestos chemicals that might have been released to the environment in the past as a consequence of the mining and milling activities at the site. The chief purpose of these data is to help refine the bounds of the study area for OU3, and to support the design of a more extensive sampling and analysis effort referred to as Phase II, which will be performed mainly in the summer and fall of 2008.

One of the goals of the RI at OU3 is to characterize exposure and risk to aquatic receptors that reside in surface water bodies that may be impacted by releases from the mined area. This includes the waters of Fleetwood Creek, Carney Creek, Rainy Creek, the on-site tailings and mill ponds, and potentially (if data indicate), the Kootenai River. Typically, water flow in these surface water features varies seasonally, being highest during the spring snowmelt period. Figure 2-2 shows the average flow pattern in a number of streams in the area as a function of time. On average, flow begins to increase around day 80 (March 20) and peaks around day 140 (May 20), although this can vary widely from year to year.

This variation in flow is potentially important because the change in flow may have significant effects on the concentrations and amounts of chemical contaminants being carried by the water. For this reason, one of the key objectives of the Phase II SAP is to collect data that will help characterize contaminant levels during the rising and falling phases of the hydrograph, as well as at other times during the summer and fall. For this reason, the Phase II sampling plan for surface water and sediment is being prepared on an accelerated schedule (ahead of the other components of the Phase II SAP), in order to ensure that sample collection can include this critical time period. The remaining parts of the Phase II SAP will be provided in Part B of the Phase II SAP.

3.0 SUMMARY OF PHASE I SURFACE WATER AND SEDIMENT DATA

3.1 Sampling Stations

During Phase I, surface water and sediment samples were collected at a total of 24 locations, as shown in Figure 3-1. As seen, sampling stations include a number of locations along Carney Creek, Fleetwood Creek, and Rainey Creek, including major ponds and impoundments on these streams, as well as seeps and springs that were located nearby.

3.2 Chemical Analyses

Surface Water

All surface water samples collected during Phase I were analyzed for asbestos, metals and metalloids, petroleum hydrocarbons, anions, and other water quality parameters. In addition, several selected surface water samples were analyzed for a broad suite of other chemicals. Table 3-1 lists the analytical methods that were employed, and Table 3-2 shows the analyses that were performed at each station.

Sediment

All sediment samples collected during Phase I were analyzed for asbestos, metals and metalloids, petroleum hydrocarbons, and several sediment quality parameters. In addition, several selected sediment samples were analyzed for a broad suite of other chemicals. Table 3-3 lists the analytical methods that were employed, and Table 3-4 shows the analyses that were performed at each station.

3.3 Results for Asbestos in Surface Water

Concentration Values

Table 3-5 summarizes the results of the analysis of surface water for asbestos (LA). Results are expressed in terms of million fibers per liter (MFL). As seen, concentration values of total LA ranged widely (more than four orders of magnitude), from < 0.1 to 125 MFL.

Figure 3-2 is a map that displays the spatial pattern of results. The highest levels were observed in samples located in ponds or impoundments, including the tailings impoundment, the mill pond, and the pond on Fleetwood creek, as well as from several seeps along the south side of the mined area. Levels in lower Rainey Creek (below the mill pond) tended to be relatively low.

Comparison to Human Health Benchmarks

Human exposure to asbestos in surface water in OU3 is likely to be occur mainly through incidental ingestion. For humans, the USEPA has identified a concentration of 7 MFL longer than 10 μm as the level of concern for drinking water. Although a few samples from OU3 exceed this level (Table 3-5, right column), none of the surface waters in OU3 are presently used for drinking water, and the amount of untreated surface water actually ingested by on-site workers or visitors is likely to be minimal.

Comparison to Ecological Surface Water Benchmarks

Although there are a number of published studies which indicated that exposure to asbestos in water can cause adverse effects on aquatic receptors, the USEPA has not yet established a Toxicity Reference value (TRV) for protection of aquatic receptors from asbestos in surface water. Therefore, no comparison of observed values to aquatic benchmarks is possible at present.

3.4 Results for Asbestos in Sediment

Concentration Values

Results for LA in sediment are expressed as mass percent (grams of asbestos per 100 grams of sediment) if the concentration is 1% or higher. If the estimated concentration is <1%, the results are expressed semi-quantitatively, according to the following scheme:

PLM-VE Result	Range of Mass Percent
A (ND)	None detected (likely < 0.05%)
B1 (Trace)	LA detected, > 0% but < 0.2%
B2 (<1%)	LA detected, >0.2% but < 1%

Table 3-6 summarize the results of the analysis of sediment asbestos (LA). As seen, nearly all (22 out of 24) of the sediment samples collected contain LA. Of these, one is classified as Bin B1 (<0.2%), 12 are classified as Bin B2 (about 0.2 to 1%), and 9 were estimated to contain levels from 2-7%.

Figure 3-3 shows the spatial pattern of LA in sediment. As shown, the only samples that are ND are those at the upper-most reaches of rainy Creek and Fleetwood Creek. High levels (Bin C) are observed in multiple locations, especially in samples collected from on-site seeps.

These results indicate that asbestos in sediment is widespread throughout the surface water features draining the site, and that levels are substantial in many locations.

Comparison to Human Health Benchmarks

The EPA has not established any guidelines for evaluating exposure of humans to asbestos in sediment. In general, risk to humans from asbestos in sediment is expected to be due mainly to inhalation exposures associated with disturbance of dried sediment, and not with incidental ingestion of sediment.

Comparison to Ecological Sediment Quality Benchmarks

The USEPA has not yet established a benchmark for protection of benthic macroinvertebrates from asbestos in sediment. Therefore, no comparison of observed values of LA in sediment to a sediment-based benchmark is possible at present.

3.5 Results for Non-Asbestos Chemicals in Surface Water

Table 3-7 summarizes the results for analytes detected in surface water samples analyzed as part of the Phase I investigation, and compares the values observed to reference values¹ for both human and ecological receptors. As seen, a number of inorganic constituents (metals, anions, and nitrogen compounds) were detected, as were several indicators of petroleum hydrocarbons, but no VOCs, SVOCs, PCBs, or PAHs were detected. None of the values detected exceeded a level of concern to humans (even if the water were used for full time drinking), and none of the values appear to suggest a substantial risk to aquatic receptors. However, as noted above, because concentration values may tend to vary over time, and may potentially be higher during spring runoff than during the fall, it is not appropriate to draw any strong risk-based conclusions from these initial data.

3.6 Results for Non-Asbestos Chemicals in Sediment

Table 3-8 summarizes the results for analytes detected in sediment samples analyzed as part of the Phase I investigation, and compares the values observed to sediment benchmark values² for both human and ecological receptors. As seen, a number of inorganic constituents were detected, as were several indicators of petroleum hydrocarbons. In addition, methyl acetate was detected in two samples, and pyrene was detected in one sample. All other chemical analytes were never detected in any sample.

Figure 3-4 summarizes the results for total extractable hydrocarbons in sediments measured by method SW8015M. As seen, nearly all samples were above the detection limit, with the highest

¹ For surface water, reference values for human health are concentrations that would pose a Hazard Quotient (HQ) of 1.0 if the water were used as a full time source of drinking water. For ecological receptors, the reference values indicate concentrations where fish or benthic macroinvertebrates may begin to display increased mortality or decreased growth or reproduction.

² For sediment, benchmark values for human health are concentrations that would pose a Hazard Quotient (HQ) of 1.0 or a cancer risk of 1E-04 if the level of exposure was the same as residential exposure to yard soil. For ecological receptors, the sediment reference values indicate concentrations where benthic macroinvertebrates may begin to display increased mortality or decreased growth or reproduction.

levels observed in the tailings impoundment, the mill pond, Fleetwood creek neat the coarse tailings pile, and in several seeps along the south side of the mined area. Perhaps unexpectedly, elevated levels were also noted in upper rainy Creek at URC-1 and URC-2. This may indicate that some of what is being detected may be from natural sources, but the data are not sufficient to draw a firm conclusion based on the Phase I data only.

Most chemicals (with the possible exception of chromium and manganese) are below a level of concern for humans even if the exposure to sediment were as extensive as to residential soil (actual human exposure to sediment in OU3 is expected to be much lower). For ecological receptors (benthic macroinvertebrates), mean and/or maximum concentration values of some metals (chromium, manganese, nickel) exceed screening benchmark values, indicating that these chemical may exceed a level of concern. However, data from only one round of sampling are too limited to draw any strong risk-based conclusions.

4.0 DATA QUALITY OBJECTIVES

4.1 Overview of the DQO Process

Data Quality Objectives (DQOs) define the type, quality, quantity, purpose, and intended uses of data to be collected (EPA, 2006). The design of a study is closely tied to its DQOs, which serve as the basis for important decisions regarding key design features such as the number and location of samples to be collected and the chemical analyses to be performed. In brief, the DQO process typically follows a seven-step procedure, as follows:

1. State the problem that the study is designed to address
2. Identify the decisions to be made with the data obtained
3. Identify the types of data inputs needed to make the decision
4. Define the bounds (in space and time) of the study
5. Define the decision rule which will be used to make decisions
6. Define the acceptable limits on decision errors
7. Optimize the design using information identified in Steps 1-6

Following these seven steps helps ensure that the project plan is carefully thought out and that the data collected will provide sufficient information to support the key decisions which must be made.

4.2 Conceptual Site Models

Figure 4-1 presents the conceptual site model for how humans may be exposed to contaminants in surface water and sediment at the site. The maximally exposed human receptor is assumed to be an area resident who hikes or fishes along site-related surface water features. Exposure is primarily via incidental ingestion of surface water or sediment, but airborne exposure may also occur by disturbance of dried sediments along stream banks. This would be of concern mainly for asbestos contamination.

Figure 4-2 presents the conceptual site model for how ecological receptors may be exposed to contaminants in surface water and sediment at the site. The maximally exposed ecological receptors are fish or benthic invertebrates that live in the streams or ponds. Wildlife (birds, mammals) may also be exposed while feeding or drinking along the streams or ponds.

4.3 Data Quality Objectives for Phase II Surface Water and Sediment Sampling

4.3.1 State the Problem

Preliminary data from Phase I indicate that surface water and sediment in OU3 are contaminated with asbestos. In addition, a number of metals and some organic chemicals have been detected. Because concentrations of chemicals in surface water tend to vary over time, especially under the influence of high flows during spring runoff, additional data are needed to characterize the sources and levels of site-related contaminants in surface water as a function of time and space. Likewise, additional data are needed to characterize the sources and levels of asbestos and possibly other contaminants in waters and sediments in OU3 drainages.

4.3.2 Identify the Decisions

Ultimately, EPA must decide if and what response actions are needed to protect human and/or ecological receptors from unacceptable risks from asbestos and any other mining-related contaminants in surface water and sediment.

4.3.3 Identify the Types of Data Needed

Contaminant Concentration Data

One type of data that is needed to evaluate risks from contaminants in surface water and sediment is reliable and representative measurements of the concentration of contaminants in surface water and sediment as a function of both time and space. This type of data is valuable both to support risk evaluations as well as to identify sources of contaminant releases.

In Phase I, the target analyte lists for surface water and sediment included not only asbestos but also a wide variety of other chemical classes (see Tables 3-1 and 3-3) in order to seek information on the occurrence of a number of potential contaminants that might have been used at the site or that might have been released from natural sources due to mining activities. As described above (see Section 3), the initial round of results support the conclusion that asbestos contamination is wide-spread in both surface water and sediment, while many of the other analytes were detected much less frequently or not at all. However, because Phase I reflects concentrations at only one point in time, these data are not considered sufficient to allow elimination of any of the analytical classes of compounds evaluated in Phase I. Therefore, all of the analytes assessed in surface water and sediment during Phase I are retained for further evaluation in Phase IIA.

Site-Specific Toxicity Tests

While measured levels of contaminants are one valuable approach for evaluating risks to exposed receptors, a second valuable approach is to expose receptors (fish, benthic macroinvertebrates) to site media (surface water, sediment) in order to observe whether the media causes adverse effects on growth, survival, and/or reproduction in laboratory test species.

Data of this type form a valuable second line of evidence that is especially useful if reliable toxicity values are not available to help evaluate measure concentration values.

For the purposes of the Phase IIA sampling plan, the medium that is most important for site-specific toxicity testing is surface water. This is because it is expected that the concentration of LA and any other site-related contaminants will be highest during spring runoff. Thus, collection and testing of water from this time interval is an essential element of any site-specific toxicity testing program. Site-specific toxicity testing will also be a key component of this program, but collection of sediment samples is not time critical and can be deferred for planning and implementation to Phase IIB.

Flow Data

Because concentrations of asbestos and other analytes in water (and also in sediment) are anticipated to depend on flow, data are needed to characterize the temporal variations in stream flow rates at numerous locations in the Rainy Creek watershed so the relation between flow and concentration may be assessed.. These data, when combined with the concentration data, will also allow an analysis of the sources and relative importance of different sources to concentration and loading in each part of the watershed.

4.3.4. Define the Bounds of the Study

Spatial Bounds

The primary focus of Part A of the Phase II investigation is Rainy Creek, Fleetwood Creek, and Carney Creek, as well as any ponds or impoundments on these streams. In addition, Phase IIA will include an evaluation of surface waters in the Kootenai River in the vicinity of the confluence with Rainy Creek, Libby Creek, and Flower Creek.

Temporal Bounds

Because surface water flow conditions are variable over time, the Phase IIA surface water investigations will be conducted during a typical range of annual flow conditions. The Phase IIA investigations will begin at the start of the rising hydrograph, and will continue through the high flow period into the summer and fall. The purpose of this temporal sampling pattern is to characterize, at least within the year 2008, the pattern of temporal variability in concentration levels of contaminants of potential concern.

4.3.5. Define the Decision Rule

The decision rules that will be used to make final risk management decisions regarding the need for remediation of surface water and/or sediment have not yet been determined. However, it is

anticipated that the rules will be based mainly on a consideration of the level of risk posed to humans and ecological receptors by site-related contaminants in surface water and sediment.

For humans, the decision rule is likely to be based on the estimated level of cancer and non-cancer risk to an individual with reasonable maximum exposure (RME). If the estimated cancer risk to the RME receptor is below some specified level (e.g., 1E-04), and if the estimated non-cancer risk is below a hazard Index of 1.0, it is likely that these site media will not be considered unacceptable for human exposure. If either the cancer or non-cancer risks exceed the maximum acceptable value, then some response action will be considered appropriate.

For ecological receptors, risk characterization will, to the extent that data allow, be based on a weight-of-evidence approach that utilizes one or more of the following strategies:

- Calculation of HQ values based on measured concentration values and available toxicity reference values (TRVs)
- Exposure of test organisms to samples of surface water and/or sediment collected from the site to evaluate the magnitude and frequency of any effects on growth or survival
- Direct surveys of receptor density and diversity in site streams in comparison to appropriate reference streams in the same area

The ecological decision rule will likely take the form that, if the weight-of-evidence indicates that adverse effects on fish and/or benthic organisms are occurring, and that these effects are likely to result in a meaningful decrease the density and/or diversity of receptors compared to what would be expected in the absence of site-related contamination, then a response action will be appropriate.

4.3.6. Define the Acceptable Limits on Decision Errors

Two types of decision errors are possible when making risk management decisions:

- A false negative decision error occurs when it is decided that risk is acceptable when the true risk is actually above the level of concern
- A false positive decision error occurs when it is decided that risk is not acceptable when the true risk is actually below the level of concern

Of these two types of errors, EPA is primarily concerned with avoiding false negative errors, since an error of this type can leave human or ecological receptors exposed to unacceptable levels of contamination and risk. The EPA usually identifies 5% as the maximum acceptable probability of making a false negative decision.

A false positive decision error does not leave humans or ecological receptors at risk, but is also of concern to EPA because this type of error may result in the expenditure of resources (time,

money) that might be better invested elsewhere. There is no Agency-wide standard for the acceptable probability of a false positive decision error, but it is common to identify as a goal that if the true level of risk is less than $\frac{1}{2}$ the acceptable risk level, then there should be no more than a 20% chance that the risk will be declared to be unacceptable.

4.3.7. Optimize the Design

The probability of making either a false negative or a false positive decision error depends on the accuracy of all of the information used to make the decision. When decisions are based on the level of computed risk, errors may occur in a number of the data items used in the computations, including the concentration term, the exposure parameters, and the toxicity term. In general, EPA seeks to limit the risk of false negative decision errors by ensuring that all uncertain inputs into risk calculations are “conservative” (i.e., are more likely to overestimate than underestimate risk). Of the uncertain inputs, the only one that is amenable to control during field sampling is the uncertainty in the concentration term. For this reason, attention in the sampling plan is focused on optimizing the number of samples that will be available for estimating average exposure levels in each exposure area for each environmental medium.

The number of samples needed to limit uncertainty in concentration term depends mainly on the nature of the underlying distribution and the degree of between-sample variability. The degree of uncertainty that can be accepted depends mainly on how close the data are to a decision criterion. That is, greater uncertainty is acceptable when the values are far removed (either below or above) the decision criterion than when the values are near a decision criterion.

In general, for non-asbestos analytes, experience has shown that, if the concentration is far removed from a decision threshold, then collection of 3-5 samples will generally be sufficient. If the concentration is close to a decision threshold, then uncertainty around the mean can be substantially reduced by collection of 10-20 samples. Collection of more than 20 samples usually does not reduce uncertainty by enough to justify the added cost.

For asbestos, uncertainty in the mean concentration arises not only from the authentic between-sample variability, but also from uncertainty in the methods used to measure the asbestos concentration. For water, concentration values for each sample are derived based on the number of fibers observed during a microscopic inspection of an aliquot of the sample. The number of fibers observed is a random variable characterized by a Poisson distribution. Because of this Poisson variation in each measured value, the overall uncertainty is a combination of the sampling variability and the measurement error, which results in a Poisson-lognormal distribution. At present, the EPA has not established a method for quantifying the uncertainty in the mean of such a data set. However, it is generally desirable to design the analysis plan such that, if fibers are present at concentration that is near the level of concern, then the number of fibers observed during an analysis will be in the 10-50 range. As above, the number of samples required depends on how close the true concentration is to the decision threshold. When

concentrations are not near the threshold, then 5-10 samples will generally be sufficient. If the concentration is close to the decision threshold, then a minimum of 10-20 samples may be required.

For sediment, the best method currently available for asbestos yields mainly semi-quantitative results. Thus, there is no statistically valid approach for deriving a quantitative estimate of the mean for a set of samples, or to characterize the uncertainty about the mean. For this reason, it is desirable to have a data set of about 10-20 samples per exposure unit in order to have a semi-quantitative understanding of spatial and potentially temporal variability of sediment levels in an exposure unit.

5.0 SAMPLING PROGRAM

All sampling of environmental media within OU3 described in this SAP will be performed by personnel who are properly trained in the field collection methods summarized in the OU3 Standard Operating Procedures (SOPs) provided in Attachment A and the Phase IIA experimental sampling design details presented below. A Health and Safety Plan (HASP) for the field sampling team will be provided by the field sampling contractor and will be reviewed by EPA and the Montana Department of Environmental Quality (MDEQ) prior to commencement of any field sampling activities.

Table 5-1 provides an overview of a number of data collection activities that will be performed under Phase IIA of the OU3 RI. Phase IIA includes additional surface water and sediment characterization within the Rainy Creek watershed, following a strategy similar to that followed in the Phase I investigations. In addition, Phase IIA includes an evaluation of surface water conditions in the Kootenai River near rainy Creek and two other tributaries that might be contributing asbestos or other contaminants to the river. The following sections present the experimental design, including sampling details and rationale, for the Phase IIA elements of surface water and sediment characterization.

5.1 Rainy Creek Watershed Monitoring – Experimental Design

A Phase I investigation within the Rainy Creek watershed was completed in the fall of 2007 to provide an initial characterization of conditions at and surrounding the Libby Mine site. Further characterization of surface water and sediment was anticipated as part of Phase II. This section describes the experimental design for Phase IIA data collection activities developed to meet data needs for surface water and sediments within the Rainy Creek watershed, as discussed above in Section 4.3.

5.1.1 Element 1: Seasonal Surface Water and Sediment Monitoring

As noted previously, it is expected that flow and concentration will vary in each portion of the Rainy Creek watershed as a function of time of year. The purpose of Element 1 is to measure stream flow and surface water and sediment concentration values at each location previously sampled in Phase I to characterize surface water and sediment during spring and summer flow conditions. These data may be combined with similar Phase I data collected during the fall of 2007 to develop a good understanding of the seasonal variability in flow and concentration patterns across the site.

Surface Water Samples

Figure 5-1 identifies the locations where samples of surface water will be collected during Phase IIA. These are the same locations where samples were collected during Phase I, plus the following additional locations:

- impoundment overflow (TP-overflow);
- Rainy Creek upstream of the mine-disturbed areas (URC-1A);
- pond on Carney Creek (CC-Pond); and
- any other additional seeps, springs, or other water features on or near the mined area not sampled during Phase I.

Table 5-2 identifies and describes all of the Phase IIA surface water monitoring locations.

All surface water samples will be single grab samples, and all samples will be analyzed for asbestos, metals/metalloids, petroleum hydrocarbons, anions, and other water quality parameters. In addition, a broad suite of analyses will be performed for samples collected at the tailings impoundment toe drain (TP-TOE1) and Lower Rainy Creek downstream of the confluence with Carney Creek (LRC-2). These locations were selected because they appear to have the best potential of characterizing releases from the mine. The additional analyses for surface water include PCBs, pesticides, herbicides, gross alpha/gross beta, volatile and semi-volatile organic compounds, and cyanide. These analyses will provide a more comprehensive screen for potential contaminants associated with mine wastes and process chemicals used during mine operations. Details on the specific chemical analyses that will be performed for surface water samples are discussed in Section 6.

Water quality data for springs will provide information on shallow groundwater quality. Seep water will provide information on whether contaminants are being released from mine waste piles and disposal areas. These data, along with any groundwater sampling data collected as part of Phase IIB, will allow for identification of mine-related contaminants and possibly an assessment of transport pathways.

At locations where flowing water is present, stream flow rate will be measured following the collection of surface water and sediment samples. Flows will be measured at locations on Fleetwood Creek, Rainy Creek, Carney Creek, at the TP-TOE1 drain and TP overflow (if running). Flow data will be used with contaminant concentration data to assess contaminant mass transport along surface water transport pathways.

Sediment Samples

Sediment samples will be collected on two separate occasions, once in late spring following peak runoff and once in late summer, from the same locations as surface water samples (see Figure 5-1). The sediment data collected during Phase IIA will be used in conjunction with data collected

during Phase I to evaluate sediment heterogeneity at each location and to assess seasonal variability in sediment conditions, if any.

The Phase IIA sediment sampling plan differs from Phase I in three main respects:

1. the tailings impoundment, which was previously sampled as part of mine waste characterization completed for Phase I, is included as a sediment sampling location for Phase IIA. As before, multiple grab samples will be collected from the near-surface materials (i.e., top 12 inches) present in the impoundment. However, rather than being composited into two samples, each of the grab samples will be analyzed individually.
2. sediment will be collected from a total of five sample locations within the Mill Pond instead of the one sample location used during Phase I; and
3. sediment will be collected from a total of five sample locations with the small pond on Carney Creek.

The purpose of collecting multiple grab samples from the tailings impoundment, the Mill Pond, and the pond on Carney Creek is to provide information on the spatial variability within each feature. This is important mainly for evaluating risks to benthic organisms in the ecological risk assessment. Additional tailings impoundment and pond sediment sampling at depth may be included in Phase IIB to characterize to characterize older materials that are now buried under the more recent mine wastes and sediment variability with depth.

A grid system will be used to guide sampling activities across the surface of the impoundment and the ponds. Figure 5-2 shows the approximate sampling locations in the tailings impoundment. [Note to EPA—we need to discuss the placement of these samples, especially regarding which are under water and which are above water] The use of a grid system provides a systematic, non-biased sampling design for evaluating the spatial variability in tailings characteristics. A total of 10 samples per event (one in late spring, one in late summer, as above) will be collected from the tailings impoundment, and five samples per event will be collected from each of the two ponds. The resulting sample density (10-20 per water body) is expected to provide data of adequate quality for use in characterization of the nature and extent of contamination and for risk assessment, as discussed in Section 4.4.7.

More detailed procedures for collection of sediment samples associated with this element of Phase IIA are given below in Section 5.3.4.

All sediment samples will be analyzed for asbestos, metals/metalloids, and petroleum hydrocarbons, and total organic carbon. Sediments from lower Rainy Creek and the tailings impoundment toe drain will also be analyzed for PCBs to assess the potential effects of use of oil for dust control along the adjacent road. Sediment collected from stations along lower Rainy Creek (LRC-2 and LRC-6) will also be analyzed for pesticides, herbicides, volatile and semi-volatile organic compounds, and cyanide. Details on the specific analyses that will be performed for sediment are discussed in Section 6.

5.1.2 Element 2: Spring Runoff Monitoring

The purpose of Element 2 is to monitor stream flow and surface water asbestos concentration values at selected stations within the Rainy Creek watershed during the rising and falling limbs of the spring-season snowmelt-runoff hydrograph. These data will be used to track changes in the asbestos content of water as stream flows first rise in response to snowmelt runoff and then decline as snowmelt ends. Surface water samples will be collected once during winter base-flow conditions, and then weekly beginning at the onset of rising stream flows in response to snowmelt, continuing through the spring high-flow season, and ending approximately 4 weeks after the seasonal peak in flow is observed on Rainy Creek.

Table 5-3 and Figure 5-3 identify the locations where samples of surface water will be collected under Element 2. Surface water samples will be collected weekly during the spring runoff season at the following locations:

- Tailings impoundment (TP), impoundment toe drain (TP-Toe1), and impoundment overflow (TP-overflow);
- Mill Pond (MP);
- Rainy Creek (URC-1A, URC-2) upstream of the mine-disturbed areas
- Fleetwood Creek (FC-2) and Carney Creek (CC-2) downstream of mine-disturbed areas
- Lower Rainy Creek below the Mill Pond (LRC-1) and below Carney Creek (LRC-2, LRC-6)
- Pond on Carney Creek (CC-Pond)

All of the locations listed were sampled during the Phase I investigation except for one new station on upper Rainy Creek (URC-1A) and one new station at a pond along lower Carney Creek (CC-Pond).

Element 2 is designed to provide a more detailed set of asbestos and flow data from a subset of the Element 1 monitoring locations. All but two of the locations selected for Element 2 are downstream of potential primary sources of asbestos, including mine waste piles and the coarse and fine tailings disposal areas, and downstream of potential secondary sources, including sediments in the Mill Pond and sediments deposited along lower Rainy Creek. The additional asbestos and flow data collected at these locations will be used to evaluate asbestos mass transport via surface water transport pathways under a range of flow conditions.

All surface water samples will be analyzed for asbestos. At locations where flowing water is present, stream discharge will be measured following the collection of surface water and sediment samples. Flows will be measured at locations on Fleetwood Creek, Rainy Creek, Carney Creek, at the TP-TOE1 drain and TP overflow (if running). Stream flows will be

measured at designated locations above and below the tailings impoundment (TP), Mill Pond (MP), and Carney Creek pond (CC-Pond) to evaluate flow-through and water residence times in these surface impoundments.

5.1.3 Element 3: Summer and Fall Monitoring

Routine Monitoring

Element 3 is an extension of Element 2 into the summer and early fall that is designed to provide ongoing information on asbestos concentrations and stream flow rates downstream of asbestos sources within the Rainy Creek watershed. Locations sampled in Element 3 are the stations on lower Rainy Creek below Carney Creek (LRC-2) and lower Rainy Creek near its discharge to the Kootenai River (LRC-6). Element 3 will start immediately upon completion of Element 2 (i.e., approximately 4 weeks after the seasonal peak in flow), and continue through September 30. Instead of weekly sample collection and flow measurement at each location as for Element 2, monitoring will be reduced to every other week for Element 3. The surface water samples will be analyzed for asbestos. Wherever flowing water is present, stream discharge will be measured following sample collection.

The two locations selected for Element 3 both represent points on the potential surface water transport pathway from mine-related sources of asbestos to lower Rainy Creek and the Kootenai River. LRC-2 is downstream of all potential mining-related sources of asbestos and downstream of two ponds representing potential secondary sources to surface water (Mill Pond and pond on Carney Creek), and LRC-6 is downstream of all potential primary and secondary asbestos sources, including sediments deposited in the lower Rainy Creek drainage downstream of LRC-2.

Storm Event Monitoring

[Note 1 to EPA reviewers: we've included storm-event monitoring as an alternative to continuous sampling and flow monitoring over the range of summer flow conditions. The proposed sampling approach will provide asbestos concentration and load data from a few short-term high-flow events, and those data can be used with similar data collected during the spring-runoff period to assess asbestos transport via surface water. We are looking for your input regarding the need for storm-event sampling in addition to the spring high-flow sampling.]

[Note 2 to EPA reviewers: Assuming we do storm event monitoring, we will be asking MWH/Remedium to develop an implementation plan for storm-event monitoring that will include equipment specifications and logistical details. The following plan is a proposal that allows some flexibility for MWH implementation. For example, the minimum size storm event used to trigger sampling represents a storm size that typically occurs numerous times during

summer/fall, however, larger storm events may actually be necessary to generate sufficient runoff to cause an increase in stream flow at the two monitored locations.]

Element 3 will also include sampling and flow measurement triggered by precipitation events that occur after the spring snowmelt-runoff season. The same two locations, LRC-2 and LRC-6, will be used for monitoring during storm events. Asbestos concentration values and flow measurements will be obtained at LRC-2 and LRC-6 to describe asbestos transport associated with three separate storm/rainfall events within the Rainy Creek drainage and to evaluate the effect of short-term, episodic surface runoff in the mine area, and related increases in local stream flow, on the asbestos content of surface water downstream of the known sources of asbestos.

Monitoring will be conducted during storm events that produce at least 0.25 inches of rainfall over a 24-hour period. Rainfall events of this minimum size typically occur in the vicinity of the Libby Mine site during summer and fall and are considered likely to occur during Phase IIA – Element 3. The stream-flow response to rainfall events of this size is not currently known because there are no detailed precipitation and flow data available to generate storm-event hydrographs for the Rainy Creek drainage. Modeling conducted by Schafer (1992) indicates that a 10-year storm event of 2.4 inches over a 24-hour period causes increased flow in local drainage approximately 12 hours following the start of rainfall. The model used by Schafer to describe the storm hydrograph was developed for a basin of undisturbed, mature forest in good condition with moderately sloped topography. The model input parameters do not match conditions in the sparsely vegetated and bare areas of steeply sloping mine waste in the mining-disturbed portion of the basin. Therefore, storm hydrographs associated with runoff from the min-disturbed areas have not been developed at this time.

A rain gage will be installed at the Libby Mine site meteorological monitoring station to provide the data needed to trigger storm-event monitoring. The rain gage will be equipped with a data logger to automatically track precipitation amounts and durations. When a rainfall event takes place, data collected at the meteorological station can be used to establish whether the event meets (or is likely to meet) the criteria given above for storm-event sampling. If so, surface water sample collection will be initiated after rising stream flow is observed in response to a qualifying storm event. Stream flow monitoring at LRC-2 may also be used to trigger storm-event-related monitoring.

The surface water sample collected at each location during each storm event will be a 24-hour, flow-weighted, composite sample. If the duration of rainfall is longer than 24 hours, additional 24-hour, flow-weighted composite samples will be collected to monitor water quality throughout the storm-related hydrograph (i.e., elevated stream flow associated with the rainfall event).

Collection of flow-weighted composite samples will require automated samplers with flow monitoring equipment. Automated samplers will be set up at LRC-2 and LRC-6 to collect the

composite samples, and a flume will be installed in the stream channel to allow for continuous flow monitoring. Each flow-weighted composite sample will be collected over a 24-hour period. The same flume and flow monitoring equipment will also be utilized for Element 4, as described below. Operation and maintenance of these automated sampling stations will be in accordance with procedures described in Section 5.3.3 and SOP Nos. 14 and 15.

5.1.4 Element 4: Continuous Precipitation and Flow Monitoring

Element 4 consists of continuous precipitation monitoring at the meteorological monitoring station at the Libby Mine site and continuous stream flow monitoring on lower Carney Creek and lower Rainy Creek. Flow monitoring will be conducted on lower Carney Creek during spring snowmelt runoff and on lower Rainy Creek during the spring snowmelt runoff, summer, and early fall. The purpose of collecting flow data on a continuous basis at these locations is to:

- characterize the spring snow-melt hydrograph while the other elements of the Phase IIA investigation are being implemented;
- track changes in flow in response to local precipitation events; and
- provide detailed flow measurements for use with asbestos concentration data to characterize asbestos mass loading to Rainy Creek from the mine site and from lower Rainy Creek to the Kootenai River.

Precipitation amounts and durations will be recorded using an 8-inch-diameter, heated, tipping-bucket rain gauge installed at the same location as the meteorological station that is currently in use at the Libby Mine site. The purpose of collecting continuous precipitation data is to determine the runoff coefficient as it relates to the precipitation intensity/duration curves for the Rainy Creek watershed. Installation and calibration of the precipitation station will be in accordance with the procedures described below in Section 5.3.5 and SOP No.17. This equipment will be installed in early spring to allow for developing relationships between precipitation and stream flow in Rainy Creek during the Phase IIA investigation.

Flumes will be installed in the stream channels at three monitoring stations: LRC-2, LRC-6, and CC-2 (see Figure 5-3). Stream flow through the flumes will be monitored using water level sensors, and the continuous flow data will be recorded using a computerized data logger. Installation and calibration of flumes will be in accordance with the procedures described below in Section 5.3.6 and SOP No. 15. The automated flow monitoring equipment will be installed before the initial rise in stream flow associated with the spring snowmelt-runoff season and that equipment will be maintained for use through the summer and early fall.

5.1.5 Element 5: Collection of Water for Toxicity Testing

As discussed in Section 4.3.3, one of the most direct methods for evaluating toxicity of site media such as surface water and sediment to ecological receptors (fish, benthic invertebrates) is

through site-specific toxicity testing. In this approach, test organisms are exposed to site media in the laboratory to determine if the site media causes adverse effects on survival, growth and/or reproduction.

In order for the results to be optimally useful, the samples of site media that are tested must include the highest levels of contaminants observed on site. If no toxicity is observed for this sample, then it is reasonable to conclude that all other samples (at lower concentrations) would also be non-toxic. If toxicity is observed, then a concentration-response curve can be developed by testing a series of dilutions of the most concentrated sample. This, in turn, can lead to the development of a site-specific TRV that can be used to predict the toxicity of other samples from the site.

At this site, it is expected that the contaminant in water most likely to be of concern to aquatic receptors is LA, and it is expected that the highest concentrations of LA in water bodies in the Rainy Creek watershed will occur at about the same time as peak flow during spring runoff. For this reason, when the hydrograph has reached an approximate maximum, a volume of water sufficient to support several series of site-specific toxicity tests will be collected from five on-site sampling stations:

- LRC-2
- LRC-4
- LRC-6
- Tailings impoundment
- Mill Pond

The sample volume collected from each station will be approximately 150 L, which shall be placed into three or 4 large plastic containers and chilled to protect against microbial growth. Each sample will be promptly submitted to the toxicity testing laboratory for toxicity testing as described in Section 7.0.

5.2 Kootenai River Monitoring – Experimental Design

5.2.1 Element 1: Kootenai River Sampling

Surface Water

Phase IIA includes two rounds of sampling of water from the Kootenai River in the vicinity of Rainy Creek to assess the effect of Rainy Creek on asbestos levels in the river water. The first sampling even will occur at approximately the time of maximum flow in Rainy Creek, and the second event will occur under summer baseflow conditions.

Sampling stations for each event will include on location (designated UKR) upstream of Rainy Creek, three stations (designated KR1, KR2 and KR3) parallel to the northern river bank downstream of the mouth of Rainy Creek, and 5 stations (designated KR4 to KR8) along a perpendicular transect downstream of Rainy Creek. The approximate locations of these stations are shown in Figure 5-4. These locations were selected to provide asbestos concentration values upstream and downstream of Rainy Creek and to include river locations with the greatest potential for elevated asbestos concentrations due to transport via Rainy Creek.

Each river-water sample will be collected from a discrete location using a depth-integrated sampler and in accordance with SOP No. 16 (Surface Water Sampling Using Depth-Integrated Samplers) and the instructions for surface water sampling in Section 5.3.1.

All samples of river water will be analyzed for asbestos.

Sediment

Phase IIA sediment sampling in the Kootenai River is more limited than in the Rainy Creek watershed because of the generally high velocity of flow in the Kootenai tends to limit the deposition of sediments. The samples that will be collected include the following:

- One grab sample from a depositional area located along the north bank of the Kootenai upstream of Rainy Creek. This will serve as a frame of reference for evaluating downstream samples.
- Two or three grab samples from a depositional areas located along the north bank of the Kootenai downstream of Rainy Creek, but within a distance of 1/2 mile.
- Two borings from the large sandbar located in the center of the river about 1/2 mile downstream. One boring will be from the highest location on the sandbar, since this may contain the oldest sediments. The other boring will be from a location near the downstream tip of the sandbar. Each boring will be to a depth that encounter the water level of the river, plus about 6 additional inches. Each boring will be subdivided into a total of four depths., or into 6-inch strata, whichever is smaller.

5.2.2 Element 2: Libby Creek Sampling

Surface water in Libby Creek has the potential to transport asbestos from sources in the town of Libby downstream to the Kootenai River. This element of Phase IIA is designed to provide preliminary characterization of asbestos levels in Libby Creek water entering the Kootenai River and the potential mass load of asbestos contributed by Libby Creek to the river.

In order to achieve this objective, data will be collected from three sampling stations: one in Libby Creek near its discharge to the Kootenai River (LC-1) and two in the Kootenai River – one

upstream (KR-9) and one downstream (KR-10) of Libby Creek. Figure 5-4 indicates the approximate locations of these samples.

Surface water samples will be collected at these three Libby Creek stations on two occasions: once during spring high-flow conditions and once during summer base flow conditions.

Surface water will be collected from Libby Creek in accordance with the grab-sampling procedures described below in Section 5.3.1 and SOP No. 3. River water samples will be collected using a depth-integrated sampling device and in accordance with SOP No. 16 (Surface Water Sampling Using Depth-Integrated Samplers) and the instructions for surface water sampling in Section 5.3.1.

Samples collected from Libby Creek and the Kootenai River will be analyzed for asbestos. Creek flow will also be measured on those two occasions, at the time of sample collection.

5.2.3 Element 3: Flower Creek Sampling

Surface water in Flower Creek has the potential to transport asbestos from sources in the town of Libby downstream to the Kootenai River. This element of Phase IIA is designed to provide preliminary characterization of asbestos levels in Flower Creek water entering the Kootenai River and the potential mass load of asbestos contributed by Flower Creek to the river.

The sampling design for Flower Creek is the same as for Libby Creek (see above). Data will be collected from one station in Flower Creek near its discharge to the Kootenai River (Flower-1) and two in the Kootenai River – one upstream (KR-11) and one downstream (KR-12) of Libby Creek. Figure 5-4 indicates the approximate locations of these samples.

Surface water samples will be collected at these three Flower Creek stations on two occasions: once during spring high-flow conditions and once during summer base flow conditions.

Surface water will be collected from Flower Creek in accordance with the grab-sampling procedures described below in Section 5.3.1 and SOP No. 3. River water samples will be collected using a depth-integrated sampling device and in accordance with SOP No. 16 (Surface Water Sampling Using Depth-Integrated Samplers) and the instructions for surface water sampling in Section 5.3.1.

Samples collected from Flower Creek and the Kootenai River will be analyzed for asbestos. Creek flow will also be measured on those two occasions, at the time of sample collection.

5.3 Field Procedures

5.3.1 Surface Water Sampling Methods and Procedures

The sampling procedures for collection of surface water grab samples are presented in OU3 SOP No. 3. During each monitoring event conducted for Rainy Creek watershed Elements 1 and 2, stream water samples will be collected from downstream to upstream locations to minimize the effect of sampling activities on the samples collected. To minimize the potential effect of time variability, all samples from a single stream drainage (i.e., Rainy Creek) will be collected on the same day. All samples will be grab samples, collected by pumping directly from the source into laboratory collection containers using a peristaltic pump. Samples will be collected from representative flowing water (usually the mid-channel).

Both filtered and unfiltered samples will be collected directly from the water bodies into sample bottles. For the filtered samples (to be analyzed for metals only), water from the source water body will be pumped through a 0.45 μm in-line, high-capacity filter using either a battery-operated peristaltic pump or hand-held manual pump. The in-line filter will be purged with approximately 200 mL of sample water before the laboratory container is filled. A new (0.45 μm) in-line filter and tubing will be used for each site to collect water for analyses of “dissolved” constituent concentrations. The filter will then be removed, and the sample for unfiltered metals and other water quality parameters will be collected.

The method for collection of water at springs, seeps, and ponds will be the same as above, except in locations of very shallow water. In such locations, water can be collected from a depression created to increase the depth of water and allow for sampling using a pump and tubing, as described in OU3 SOP No. 3.

5.3.2 Surface Water Field Measurements and Flow Monitoring

Whenever grab samples of surface water are collected, the in-stream temperature, pH, specific conductance, dissolved oxygen (DO), and turbidity will also be measured using portable field meters. Field parameter measurement and calibration protocols will be performed according to manufacturer’s specifications and OU3 SOP No. 10. These measurements will be recorded on field sampling forms.

At locations where flowing water is present, stream discharge will always be measured following the collection of surface water and sediment samples. The stream flow will be measured and recorded in accord with OU3 SOP No. 4. In brief, discharge will be measured using one of three portable methods, as dictated by flow or channel characteristics. Depending on the channel characteristics and flow, an area-velocity method, a portable flume, a volumetric method, or some combination of these methods, will be used to obtain the stream discharge measurements. Field personnel responsible for stream-discharge measurements must have prior experience using the methods and equipment described in OU3 SOP No. 4.

In cases where water depth is greater than 0.3 feet or the channel cross section is wide, flow generally will be measured using the area-velocity method of stream-flow gauging as described in the National Handbook of Recommended Methods for Water Data Acquisition (USGS, 1977), and explained in detail in OU3 SOP No. 4. Using this method, the stream cross section is divided into a series of subsections where the average depth, average velocity, and width for the subsections are measured.

A portable cutthroat flume will be used to measure flow when low discharge and/or channel geometry preclude the use of a velocity meter. The flume will have a throat width adjustable from 2 to 8 inches, which can be used to measure flows from approximately 0.01 to 2.2 cfs. All water will be routed through the leveled flume, to the extent practicable, after which the height (to the nearest 0.01 foot), throat width, and leakage estimate as a percentage (if any) will be recorded. Discharge will be calculated using these data and an equation that is specific to the flume size.

In cases where flows are too small or stream gradients are too great to be measured using the area-velocity method or a cutthroat flume, measurements will be made volumetrically using a calibrated collection container and a stopwatch. Stream flow will be routed through a PVC pipe and the time to fill a collection container to a known volume will be measured. A minimum of five trials will be executed for each volumetric measurement, and discharge will be taken as an average of the five trials. An estimate of any leakage around the routing pipe will be recorded.

5.3.3 Automated Sampler Specifications and Procedures

The automated sampler chosen for this application must be capable of creating flow-derived composite samples. Therefore it is crucial that the pressure transducer in the flume and the automated sampler are compatible. The flow-derived composite sample can be created by varying the aliquot volume at a constant time interval or by varying the time interval and keeping the aliquot volume constant. Either method should be adequate to determine contaminant loading rates during storm runoff.

The automated sampler will be located out of the floodplain, on relatively level ground, but not above the suction head capacity of the automated sampler pump. Additionally, the intake line to the sampler will be kept as short as possible to minimize cross-contamination of the samples and the intake installed upstream or within the approach to the flume. It may be necessary to place the sampler in a securable enclosure if extreme weather and/or vandalism are reasonably anticipated. If connection to a power source is available, the automated sampling station will operate on 120VAC.

Routine maintenance of the automated sampler will be completed during each visit. The unit will be checked for faults, errors, or alarms during the previous sampling interval and the associated issues will be resolved. It will be determined if adequate sample volumes were collected and

reprogramming of the sampling interval/volume will be completed if necessary. After each sampling event, the pump tubing will be inspected for wear and replaced if necessary. The intake line will be cleaned and the intake foot will be inspected to verify that is not buried in newly deposited sediment or plugged by debris.

Additional detailed instructions for the operation and maintenance of automated sampling stations is provided in SOP No. 14.

5.3.4 Sediment Sampling Methods and Procedures

At each sampling location, sediment will be collected in accord with OU3 SOP No. 5. In brief, a single sediment sample will be collected from each station. Each sample will consist of a composite of five grab samples collected from low-energy (i.e., depositional) portions of the stream channel that are inundated by creek water at the time of sampling (i.e., locations of sediment deposition to channel). The five grab samples will be collected over a reach that is within 100 feet upstream or 100 feet downstream of the specified station. Each grab sample will be collected using the “direct sampling” method and compositing instructions included in OU3 SOP No. 5. The mass of sediment collected may be estimated by visual assessment of sediment volume. If the mass of sediment from the inundated areas is not sufficient for the analyses that are required (refer to Section 5.5 below), sediment will be collected from within the active high-flow channel, but no sediments will be collected from over-bank areas. After homogenization, the composite sample may be split to fill appropriate containers for the analyses requested.

All sampling and field measurement equipment that is used at more than one sample station must be decontaminated following each use. Appropriate equipment decontamination procedures are provided in OU3 SOP No. 7.

5.3.5 Precipitation Monitoring

The precipitation monitoring station will be installed at the same location as the existing meteorological station at the Site. This monitoring station will be equipped with a wind shield for better accuracy as well as 2 heaters maintained at 40°F (one on the collector funnel and one on the drain tube) to allow for measurement of precipitation under freezing conditions. The gauge will be compatible with the power supply and the data-logger/data-transmission system that are currently used at the meteorological station. The gauge will have a resolution of 0.01 inches with a range of 0-10 inches per hour. The accuracy will be at least ± 0.02 inches or 4% of the hourly total (whichever is greater). The gauge will log or transmit the date and time for each tip (0.01 inches) so that duration/intensity curves can be derived from the data collected.

The rain gauge will be installed in an area that is representative of the Rainy Creek watershed within a reasonable distance (i.e., <100yds) from the existing meteorological station. The instrument will be calibrated after installation and the calibration will be checked at least once

every three months thereafter. Installation, calibration, and maintenance procedures are provided in OU3 SOP No.17.

5.3.6 Continuous Flow Monitoring with Data Logger

The continuous flow monitoring station will be capable of measuring flows from 1 to 150 cfs. This flow range should be adequate to measure a typical spring snowmelt event as well as base-flow conditions, but it may not be large enough to measure very large storm events such as the predicted 10 year, 24-hour storm (Schaffer 1992). A nested Parshall flume will be used for this sampling system due to their ability to accurately measure a large range of flows. The flume design will have two stilling wells to accommodate automated water-level measurements even under submerged outlet conditions. Data logging pressure transducers will be secured in the stilling wells so that they can be used to measure and record the water levels within the flume on a 15 minute interval. The pressure transducer will also be capable of communicating with an automated sampler to allow for collection of flow-weighted composite samples.

Proper installation of the flume is critical to obtaining accurate measurements. Installation, calibration, and maintenance of flumes are discussed in this Section with more detail provided in SOP No. 15.

Ideally, the installation location should have a relatively mild slope leading up to the flume and a steep slope (or drop off) after the flume. The flume must be bedded and secured to a concrete base ensuring that the throat is level both laterally and longitudinally, the sides are vertical, and to prevent shifting of the flume during high flow conditions. The structures constructed to divert all flow through the flume must be robust enough to withstand complete submergence during large storm events without degradation. They must also be made of materials that will minimize seepage around or under the flume.

Maintenance of the flume includes removal of debris from the approach, checking for seepage around the flume, cleaning sediment from the base of the flume, cleaning the stilling well ports, and checking the calibration of the pressure transducers. This maintenance should be completed on a routine basis as well as after large storm events.

5.3.7 Field Documentation

Field documentation procedures are described in Section 5.5 and OU3 SOP No. 9. Field documentation associated with surface water and sediment sampling will also contain information of sufficient detail to fully describe:

- sample depth (sediment),
- sampling method, and

- associated field measurements, including stream discharge if measured, and field measurement methods.

Field measurement values are generally reported directly in the units of final use in the field notebook and data sheets without need for additional calculations (e.g., pH, temperature, and conductivity measurements). The field data will be reviewed daily by the field supervisor to identify anomalous data and transcriptional and/or computational errors. Corrective actions will be initiated as appropriate; these actions may consist of re-measuring a particular parameter, collecting a new sample, or other applicable corrective action measures.

5.4 Sample Handling Instructions

5.4.1 Sample Containers

All sample containers used for sample collection and analysis for this project will be prepared according to the procedures contained in the EPA document, *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers*, dated December 1992. This document specifies the acceptable types of containers, the specific cleaning procedures to be used before samples are collected, and QA/QC requirements relevant to the containers and cleaning procedures. The analytical laboratories will supply all sample containers utilized for this investigation. If field personnel observe any cracked or dirty containers, or if the appropriate preservative is missing in the sample bottles, those containers will be discarded and the laboratory will be notified of the problem to prevent its re-occurrence.

Tables 5-4 and 5-5 identify the appropriate sample containers for the analysis methods used in Phase IIA for surface water and sediment samples, respectively.

5.4.2 Sample Preservation and Storage

Tables 5-4 and 5-5 describe the sample preservation and storage requirements for solid and aqueous media, respectively. Samples will be preserved using appropriate preservatives in order to prevent or minimize chemical changes that could occur during transit and storage. Solid samples (soil and sediment) typically do not require preservation other than temperature control during storage and transfer to the laboratory. The exception is solid samples collected for analyses of volatile organic compounds, including VPH and TCL VOCs. Soil and sediment samples collected for analysis of VPH and TCL VOCs will be preserved in the field with methanol based on EPA SW-846 method 5035.

5.4.3 Sample Holding Times

A holding time is defined as the allowable time between sample collection and analysis and/or extraction recommended to ensure accuracy and representativeness of analysis results, based on

the nature of the analyte of interest and chemical stability factors. The holding time is calculated from the date and time of sample collection to the time of sample preparation and/or analysis. Sample holding times are established to minimize chemical changes in a sample prior to analysis and/or extraction. Samples will be shipped to the laboratory as soon as possible after collection or processing. There are currently no EPA guidelines for holding times for solid samples analyzed for metals/metalloids and most other inorganic constituents, but a six-month holding time is recommended. There is not holding time requirement for asbestos.

Tables 5-4 and 5-5 define method-specific analytical holding times for solid and aqueous media, respectively.

5.4.4 Sample Archival and Final Disposition

Unused samples and containers will be maintained in storage at the laboratory for a minimum of 90 days following completion of the analysis, unless otherwise directed by EPA. After 90 days or approval from EPA for disposal, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods, and will have disposal company contracts on file for inspection.

All data generated during the analysis of project samples must be stored by the laboratory for a period of ten years. Revised copies of the applicable SOPs and QAPPs must also be maintained and available should the data be required.

5.5 Sample Documentation and Identification

Data regarding each sample collected will be documented in accord with OU3 SOP No. 9 using Libby-specific field sample data sheets (FSDS). Any special circumstances that influence sample collection or result in deviations from sampling SOPs will be documented in a field log book.

At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number. The sample ID for all samples collected as part of Phase II (including both Phase IIA and IIB) sampling activities will have a prefix of “P2” (e.g., P2-12345). Information on whether the sample is representative of a field sample or a field-based quality control (QC) sample (e.g., field blank, field split) will be documented on the FSDS, but this information will not be included on the chain-of-custody to make certain that the sample type is unknown to the analytical laboratory.

Each field sampling team will maintain a field log book. The log book shall record all potentially relevant information on sampling activities and conditions that are not otherwise

captured on the FSDS forms. Examples of the type of information to be captured in the filed log include:

- Names of team members
- Current and previous weather conditions
- Field sketches
- Physical description of the location relative to permanent landmarks
- Number and type of samples collected
- Any special circumstances that influenced sample collection

As necessary for sample collection and location documentation, photographs will be taken using a digital camera. GPS coordinates will be recorded for all sampling locations on the FSDS form. A stake or pole identifying the sampling station will be placed at or near the sampling station for future identification of the location.

5.6 Sample Chain of Custody and Shipment

Field sample custody and documentation will follow the requirements described in OU3 SOP No. 9. Sample packaging and shipping will follow the requirements described in OU3 SOP No. 8.

A chain-of-custody form specific to the Phase IIA OU3 sampling shall accompany every shipment of samples to the analytical laboratory. The purposes of the chain-of-custody form are: a) to establish the documentation necessary to trace possession from the time of collection to final disposal, and b) to identify the type of analysis requested. All corrections to the chain-of-custody record will be initialed and dated by the person making the corrections. Each chain-of-custody form will include signatures of the appropriate individuals indicated on the form. The originals will accompany the samples to the laboratory and copies documenting each custody change will be recorded and kept on file. One copy of the chain-of-custody will be kept by field personnel.

All required paper work, including sample container labels, chain-of-custody forms, custody seals and shipping forms will be fully completed in ink (or printed from a computer) prior to shipping of the samples to the laboratory. Shipping to the appropriate laboratory from the field or sample storage will occur through overnight delivery.

All samples that may require special handling by laboratory personnel to prevent potential exposure to LA or other hazardous substances will be clearly labeled.

Upon receipt, the samples will be given to the laboratory sample custodian. The shipping containers will be opened and the contents inspected. Chain-of custody forms will be reviewed for completeness and samples will be logged and assigned a unique laboratory sample number.

Any discrepancies or abnormalities in samples will be noted and the Project Manager or the appropriate delegate will be promptly notified.

Chain-of-custody will be maintained until final disposition of the samples by the laboratory and acceptance of analytical results.

6.0 LABORATORY ANALYSIS REQUIREMENTS

6.1 Analytical Methods for Asbestos

All laboratories that analyze samples of surface water or sediment for asbestos as part of this project must participate in and have satisfied the certification requirements in the last two proficiency examinations from the National Institute of Standards and Technology/National Voluntary Laboratory Accreditation Program (NVLAP). Laboratories must also have demonstrated proficiency by successful analysis of Libby-specific performance evaluation samples and/or standard reference materials, and must participate in the on-going laboratory training program developed by the Libby laboratory team.

6.1.1 Surface Water

All surface water samples collected during Phase IIA sampling will be submitted for asbestos analysis using transmission electron microscopy (TEM) in accord with the International Organization for Standardization (ISO) 10312 method (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications, including the most recent versions of modifications LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000053, and LB-000066 (see Attachment D). An aliquot of water (generally about 100 mL) will be filtered through a 47 mm mixed cellulose acetate (MCE) filter with pore size of 0.2 μm , using a backing filter with pore size of 5 μm . All amphibole structures (including not only LA but all other amphibole asbestos types as well) that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length greater than or equal to 0.5 μm and an aspect ratio (length:width) > 3:1, will be recorded on the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheets ("TEM Water EDD.xls"). Data recording for chrysotile, if observed, is not required.

The target analytical sensitivity for asbestos in water is 50,000 f/L (50 f/mL). The human health maximum contaminant level (MCL) for asbestos in drinking water is 7,000,000 f/L and is based on fibers longer than 10 μm in length. Upon review of available ecological toxicity data in the literature, it appears that effects thresholds range from about 10,000-1,000,000 f/L for aquatic receptors and wildlife. Therefore, a target analytical sensitivity of 50,000 f/L should be adequate to provide screening level risk estimates for humans and most ecological receptors of interest. This sensitivity can be achieved by filtering 100 mL of water and counting about 20 GOs., assuming that filter overloading does not occur.

Stopping rules for these analyses are as follows:

1. Calculate the number of GOs needed to achieve the target sensitivity.

2. If the target sensitivity can be achieved by counting 50 or fewer GOs, count until the target sensitivity is achieved, or until 50 LA structures are observed. If 50 LA structures are observed, finish counting the GO containing the 50th structure, then stop.
3. If the target sensitivity requires more than 50 GOs, count until 50 GOs are counted, or until 50 LA structures are observed. If 50 LA structures are observed, finish counting the GO containing the 50th structure, then stop.

6.1.2 Sediment

Sample Preparation

All sediment samples collected for asbestos analysis will be transmitted to the CDM soil preparation laboratory in Denver, Colorado. Samples will be prepared in accordance with ISSI-LIBBY-01 Revision 10. In brief, the raw sediment sample is dried and then split into two aliquots. One aliquot is placed into archive, and the other aliquot is sieved into coarse (> ¼ inch) and fine fractions. The fine fraction is ground to reduce particles to a diameter of 250 um or less and this fine-ground portion is split into 4 aliquots.

Sample Analysis

Each sediment sample will be analyzed for LA in accordance with Libby site-specific SOPs. The coarse fraction (if any) will be examined using stereomicroscopy, and any particles of LA will be removed and weighed in accordance with SRC-LIBBY-01 Revision 2. One of the fine ground fraction aliquots will be analyzed by polarized light microscopy (PLM) using the visual area estimation method (PLM-VE) in accordance with SRC-LIBBY-03 Revision 2. Mass fraction estimates and optical property details will be recorded on the Libby site-specific laboratory bench sheets and EDD spreadsheets.

6.2 Analytical Methods for Other (Non-Asbestos) Analytes

This section describes the laboratory analysis methods selected to provide non-asbestos chemical data to support the Phase IIA data quality objectives. Methods employed are derived from the following sources:

- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods* (EPA, 1986)
- *Methods for Chemical Analysis of Water and Wastes* (EPA, 1994b)
- Montana Department of Environmental Quality method specifications for petroleum hydrocarbons (MDEQ, 2003)

Detailed calibration procedures and quality control practices associated with each referenced method are described later in Section 8.

The laboratories performing chemical analyses will be required to follow procedures for each referenced method in accordance with the method protocols in the original source documents. All method-specific quality control measures, such as external and internal standard calibration procedures, instrument performance verifications, and quantitation using method of standard additions, specified within any referenced EPA method number will be performed.

6.2.1 Water

Non-asbestos analyses required for surface water samples are listed in Table 6-1. Analytes included under each method are identified in Table 3-1.

[Note to EPA--What RfDs will we use to evaluate human health risk from hydrocarbons? The main exposure pathway would presumably be oral. The Montana values are stated to be "risk based", but the basis of the risk-based levels is not clear. We need to be sure that our analytical method is consistent with our risk evaluation approach.]

6.2.2 Sediment

Non-asbestos analyses required for surface water samples are listed in Table 6-2. Analytes included under each method are identified in Table 3-3.

6.3 Instrument Calibration and Frequency

All laboratory instruments used in the analysis of samples generated during this project must be calibrated by the laboratory in accordance with the requirements of the instrument manufacturer and the requirements specified in the relevant analytical method. Calibration records will be kept in logbooks for all instruments. It is the responsibility of the Laboratory Quality Assurance (QA) Officer to assure that calibration data is properly logged in the logbooks for each analysis.

6.4 Laboratory Custody Procedures and Documentation

The laboratories will implement the following procedures:

- A sample custodian will be designated.
- Upon receipt at the laboratory, each sample shipment will be inspected to assess the condition of the shipping container and the individual samples.
- Enclosed chain-of-custody records will be cross-referenced with all the samples in the shipment. These records will be signed by the sample custodian and placed in the project file.

- Sample storage will be secured (in the appropriate environment, i.e., refrigerated, dry, etc.), sample storage records and intra-laboratory sample custody records will be maintained, and sample disposal and disposal date will be properly documented.
- Internal chain-of-custody procedures will be followed by assigning a unique laboratory number to each sample on receipt; this number identifies the sample through all further handling;
- Internal logbooks and records will maintain the chain of custody throughout sample preparation and analysis, and data reporting will be kept in the project files.
- The original chain-of-custody record will be returned to the Project QA Officer with the resulting data report from the laboratory.

It is the laboratory's responsibility to maintain internal logbooks and records throughout sample preparation, analysis, and data reporting.

6.5 Laboratory Health and Safety

All laboratories analyzing samples from OU3 must be properly trained in the safe handling, storage and disposal of samples that may contain LA and other potentially hazardous materials.

6.6 Documentation and Records

Data reports will be submitted to the Project Manager and include a case narrative that briefly describes the number of samples, the analyses, and any analytical difficulties or QA/QC issues associated with the submitted samples. The data report will also include signed chain of custody (COC) forms, analytical data summary report pages, and a summary of laboratory QC sample results and raw data, where applicable. Raw data are to consist of instrument preparation and calibration logs, instrument printouts of field sample results, laboratory QC sample results, calibration and maintenance records, COC check in and tracking, raw data count sheets, spectra, micrographic photos, and diffraction patterns.

6.7 Data Deliverables

Asbestos data generated during this project will be entered into Libby-specific EDD spreadsheets by appropriately trained data entry staff. The data to be captured will include all relevant field information regarding each environmental sample collected, as well as the analytical results provided by the laboratory. Analytical results will include the structure-specific data for all TEM analyses and optical properties data for all PLM analyses. All data entry will be reviewed and validated for accuracy by the laboratory data entry manager or appointed delegate.

Non-asbestos data generated for this project will be transmitted via an EDD spreadsheet. The specific structure and format of this spreadsheet will be specified by the project data manager

and will be provided to the laboratory for data submittal. All data entry will be reviewed and validated for accuracy by the laboratory data entry manager or appointed delegate.

All asbestos and non-asbestos EDDs will be submitted to EPA technical contractors (SRC) electronically. Whenever possible, data files should be transmitted by e-mail to the following address:

LibbyOU3@syrres.com

When files are too large to transmit by e-mail, they should be provided on compact disk to the following address:

Lynn Woodbury
Syracuse Research Corporation
999 18th Street, Suite 1975
Denver CO 80202

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the Project Manager. At the termination of Phase I, all original data records will be provided to the EPA Project Manager for incorporation into the OU3 project files.

7.0 TOXICITY TESTING REQUIREMENTS

Toxicity tests using site-specific surface waters will be conducted in a phased approach, as follows.

7.1 Tests Using Undiluted Water

As described above, large volume samples of water will be collected from seven different locations at the site at approximately the peak of the spring runoff event. Each of these seven waters will be tested using SOP-xxx. In brief, the test organism will be rainbow trout fry [Note to EPA: OK? Need to test a benthic organism? If so, need more water?] The test system for each sample will consist of three 4-L aquaria, each housing 15 fry. Water in each aquarium will be changed once per week. Exposure will continue for 4 weeks (28 days). Air bubblers will be used to minimize settling of fibers. Endpoints measured during the study will include qualitative observations of effects of exposure on swimming and feeding behavior, as well as quantitative data on mortality and growth.

7.2 Tests Using Diluted Water

If toxicity is observed in any of the samples evaluated as above, the next step will be to perform toxicity tests on a dilution series prepared from the most toxic water sample. The dilutions will be 100% (undiluted), 50%, 25%, 12%, 6% and 3%. Each test will use the same protocol as described above, in accord with SOP-xxx.

7.3 Tests Using Asbestos-Spiked Water

If no toxicity is observed in any of the five undiluted site-specific samples described in Section 7.1, a spiking study will be performed to confirm the results and extend the observations to an even higher water concentration. The water used in the spiking test will be moderately hard reconstituted laboratory water, and the spiking material will be fibers of LA derived from ore at the Libby mine site. Spiking levels will be adjusted to be about 50%, 100%, 200%, 300% and 400% of the highest values measured in the site waters.

7.4 Data Reporting

For each water sample tested, the laboratory shall record data using the data sheet provided in Table 7-1. The laboratory shall also provide a text report in which the conditions of the test and any deviations from the SOP are presented.

8.0 QUALITY CONTROL

Quality Control (QC) is a component of the QAPP, and consists of the collection of data that allow a quantitative evaluation of the accuracy and precision of the field data collected during the project. QC samples that will be collected during this project include both field-based and laboratory-based QC samples.

8.1 Field-Based Quality Control Samples

Field-based QC samples are those samples which are prepared in the field and submitted to the laboratory in a blind fashion. That is, the laboratory is not aware the sample is a QC sample, and should treat the sample in the same way as a field sample. In general, there are three types of field QC sample: blanks, field splits/duplicates, and performance evaluation (PE) samples. Table 8-1 summarizes the types and frequency of field QC samples which will be collected during Phase IIA.

8.1.1 Blanks

Field Blanks

A field blank is a sample of the same medium as field samples, but which does not contain any contaminant. Field blanks are collected for water samples, but not for sediment.

A field blank for water shall be prepared by placing an appropriate volume of analyte-free reagent water (e.g., ASTM Type II) into a sample collection container. Field blanks for water will be collected at a rate of at least 10% (1 field blank per 10 field samples, or 1 per sample batch, whichever is greater).

Trip Blanks

The trip blank is used to indicate potential contamination of field samples by VOCs during sample shipping and handling. A trip blank consists of analyte-free laboratory reagent water which accompanies the empty sample bottles to the field and is placed in each cooler containing samples scheduled for VOC analysis. The trip blank is not opened until analysis in the laboratory with the corresponding site samples.

During Phase IIA sampling, one trip blank per cooler will be prepared to accompany aqueous samples when they are shipped to the laboratory for VOC analysis. One trip blank per cooler will also be prepared to accompany solid samples shipped for analysis of EPH and VPH.

Equipment Rinsate Blanks

Equipment rinsate blanks determine if decontamination procedures of field equipment are adequate to prevent cross-contamination of samples during sample collection. An equipment rinsate blank is prepared by rinsing decontaminated field equipment with analyte-free reagent water. Equipment rinsate blanks will be collected at a rate of 1 per sampling team per day. If field equipment is not re-used between sampling locations (i.e., dedicated equipment is used or equipment is disposable and decontamination is not necessary), equipment rinsate blanks will not be collected.

8.1.2 Field Splits/Duplicates

A field split is a sample that is prepared by thoroughly homogenizing a field sample, dividing the homogenized sample into two parts, and analyzing each independently. A comparison of field split samples is a measure of the precision of the sample preparation and analysis methods.

A field duplicate is a field sample that is collected at the same place and time as an original field sample. However, because of potential variation in field duplicate samples (even those from similar locations, especially for media such as soil, waste rock, tree bark, sediment, etc.), it is not appropriate to assume that field duplicate pairs must necessarily have the same or similar concentration values. Rather, field duplicates help to evaluate variability due to small-scale media heterogeneity, along with analytical precision.

Table 8-1 summarizes the frequency that field splits and duplicates will be collected for each media. In general, field splits/duplicates will be prepared at a rate of approximately 10% (1 field split/replicate per 10 field samples). The exception will be solid media samples collected within the mined area. There are four types of solid media that will be collected within the mined area – mine waste, roadway materials, coarse tailings, and fine tailings. Within the mined area, 1 field duplicate will be collected for each type of solid media. The specific stations at which field splits/duplicates will be collected will be determined in the field based on sampling conditions.

8.1.3 Performance Evaluation (PE) Samples

Performance Evaluation (PE) samples are samples of a matrix that contain a known and certified level of a contaminant. The results of PE sample analysis help evaluate analytical accuracy. PE samples for water and in soil are available through the EPA Quality Assurance Technical Support (QATS) program. A total of 4 water PE samples and 3 soil PE samples containing a range of inorganic and organic analytes will be added in random order to the field samples by the field collection teams.

PE samples for LA in soil are available from USGS. These PE samples were prepared by mixing uncontaminated soil samples from Libby with known amounts of LA collected from the mine, so the true mass fraction of LA is known. A total of 4 PE samples representing a range of LA

levels will be added in random order to the field soil samples at the time of soil sample preparation.

8.2 Laboratory-Based Quality Control Samples for Asbestos Analysis by TEM

The QC requirements for TEM analyses of air samples at the Libby site are patterned after the requirements set forth by NVLAP. There are three types of laboratory-based QC analyses that are performed for TEM. Each of these is described in more detail below.

Lab Blank - This is an analysis of a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

Recounts - A recount is an analysis where TEM grid openings are re-examined after the initial examination. The type of recount depends upon who is performing the re-examination. A *Recount Same* (RS) describes a re-examination by the same microscopist who performed the initial examination. A *Recount Different* (RD) describes a re-examination by a different microscopist within the same laboratory than who performed the initial examination. An *Interlab* (IL) describes a re-examination by a different microscopist from a different laboratory.

Repreparation - A repreparation is an analysis of a TEM grid that is prepared from a new aliquot of the same field sample as was used to prepare the original grid. Typically, this is done within the same lab as did the original analysis, but a different lab may also prepare grids from a new piece of filter.

As described the most recent Libby-specific Laboratory Modification #29 (LB-000029 in Attachment D), lab blanks will be performed at a frequency of 4%, recounts will be performed at a frequency of 5%, and repreparations will be performed at a frequency of 1%. LB-000029 summarizes the project-specific acceptance criteria for TEM QC analyses for all participating laboratories.

8.3 Laboratory-Based Quality Control Samples for Asbestos Analysis by PLM

8.3.1 Preparation Laboratory QC Samples

Soil Preparation QC samples are collected to ensure proper sample handling and decontamination of soil preparation equipment. Preparation QC samples are assigned unique field identifiers and are submitted blind to the analytical laboratory along with the field samples. Thus, the analytical laboratories cannot distinguish field samples from preparation QC samples. Two types of preparation QC samples are included for PLM analysis. Each of these is described in more detail below.

Preparation Blank – A preparation blank consists of asbestos-free quartz sand which is processed with each batch of field samples. A batch of samples is defined as a group of samples that have been prepared together for analysis at the same time (approximately 125). Preparation blanks determine if cross-contamination is occurring during sample preparation processing (i.e., drying, sieving, grinding, and splitting). The target number of preparation blanks is 1 per batch. All preparation blanks shall be PLM-VE Bin A (non-detect). If a preparation blank is ranked as a detect, the procedures for equipment decontamination between samples will be revised and revised as needed.

Preparation Splits – Preparation splits are prepared by dividing a sample into two parts after drying but prior to sieving and grinding. One preparation duplicate is included for every 20 field samples prepared. Because preparation splits may be authentically different due to within-sample heterogeneity, there are no acceptance criteria for preparation splits. Comparison of the results for preparation splits with the paired original field samples helps to evaluate the variability that arises during the preparation and analysis steps.

8.3.2 Analytical Laboratory QC Samples

As part of PLM-VE analysis, laboratory duplicate analyses will be prepared at a frequency of 10% (1 per 10 analyses). A *laboratory duplicate* is a re-preparation of a soil sample slide by a different analyst than who performed the initial analysis. Laboratory duplicates are performed to evaluate potential analytical differences between analysts. The acceptance criterion for laboratory duplicate analyses is that no more than 10% of all samples shall be discordant (assigned different PLM-VE bins). If the discordance rate is greater than 10%, laboratory procedures for sample examination and bin-assignment shall be reviewed and staff re-trained, as needed.

8.4 **Laboratory-Based Quality Control Samples for Non-Asbestos Analyses**

The following subsections describe laboratory-based quality control measures used to assess and document the quality of analytical results for non-asbestos parameters. Laboratory QC sample analysis frequencies and control limits used by contracted laboratories will be in accordance with referenced analytical method protocols, and the QC analyses and results will be documented and reported to EPA by the selected laboratory.

Table 8-2 summarizes all laboratory quality control measures, control limits, and corrective actions for this project, by analysis method. All laboratory QC data will be reported with results of associated sample analyses to allow for comparison of QC results to the QC criteria specified for this project.

8.4.1 Method Blank

Method blanks are designed to measure laboratory-introduced contamination of environmental samples. Method blanks verify that method interferences caused by airborne contaminants, solvents, reagents, glassware, or other sample processing hardware are known and minimized. The blank will be ASTM Type II water (or equivalent) for water samples. The method/reagent blank is processed through all procedures, materials, and lab-ware used for sample preparation and analysis.

The frequency for method blank preparation and analysis is a minimum of one per twenty field samples or per analytical batch, whichever is most frequent. An analytical batch is defined as samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch are to be of similar composition or matrix.

Acceptance criteria and corrective action for out-of-control method blanks are provided in Table 8-2.

8.4.2 Laboratory Control Samples

Laboratory control samples (LCSs) are designed to check the accuracy of the analytical procedure by measuring a known concentration of an analyte of interest. LCS samples are prepared by spiking clean, laboratory-simulated matrices (reagent-free water or purified solid matrix) with representative analytes at known concentrations that are approximately 10 times greater than the method's quantitation limits. These spiked samples are then subjected to the same preparation and analytical procedures as associated environmental samples. A LCS will be analyzed with every analytical batch, and the measured concentrations will be compared to the known, or spiked, concentrations of the LCS to compute a percent recovery value.

LCSs will be analyzed at a minimum frequency of one per every 20 samples or one per analytical batch of no more than 20 samples. Control limits for laboratory control samples are listed on Table 8-2. Failure of the LCS to meet recovery criteria requires corrective action before any further analyses can continue.

For some methods, a duplicate of the LCS is also analyzed with each analytical batch and the difference between the LCS and the LCS Duplicate (LCSD) indicates the precision of laboratory sample preparation and analysis methods at a known concentration level. Control limits for precision measured by the RPD of LCS/LCSD results are listed in Table 8-2. When LCSD samples are analyzed, the minimum frequency of analysis is one per every 20 samples.

8.4.3 Matrix Spikes/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) samples are designed to evaluate the effect of the sample matrix on analytical data, by measuring precision and accuracy from a known concentration of a target analyte that has been added to a particular sample matrix. MS/MSD samples are prepared by spiking environmental field samples with a standard solution containing known concentrations of representative target analytes. The MS/MSD sample pair is prepared from three volumes of an environmental sample. Two portions of the sample (the MS and the MSD) are spiked with the standard solution. The remaining volume is not spiked. The spiked samples are analyzed, and the percent recovery (PR) and relative percent difference (RPD) between the results of the MS analysis and the MSD analysis are calculated. The unaltered sample volume is analyzed as an ordinary environmental sample.

Sampling personnel will identify for the laboratory which samples are to be used for MS/MSD preparation. Field blanks and field duplicates are not used as MS/MSDs. Typically, additional sample volume will be required to prepare the MS and MSD, especially for analyses of water samples for organic compounds. MS/MSDs will be analyzed at a minimum frequency of one per every 20 samples.

Background and interferences that have an effect on the actual sample analyte will have a similar effect on the spike. The calculated percent recovery of the matrix spike is considered to be a measure of the relative accuracy of the total analytical method, i.e., sample preparation and analysis. The matrix spike is also a measure of the effect of the sample matrix on the ability of the methodology to detect specific analytes. Acceptance criteria and corrective action procedures for out-of-control matrix spike results are listed in Table 8-2.

8.4.4 Surrogate Spike Analyses

Surrogate spike analyses are used to determine the efficiency of target analyte recovery during sample preparation and analysis. A surrogate spike is prepared by adding a known amount of surrogate compound to an environmental sample before extraction. The surrogate compound is selected to exhibit an analytical response that is similar to the response displayed by a target compound during sample analysis. The accuracy of the analytical method is measured using the calculated percent recovery of the spiking compound. Poor reproducibility and percent recovery during surrogate spike analyses may indicate sample matrix effects.

Surrogate compounds are not added to inorganic analyses; however, surrogates are required for most organic analyses. Both environmental and QC samples are spiked with surrogate compounds. Surrogate spike recoveries are acceptable if the results of a surrogate spike fall within the control limits established by laboratory QC protocol. Acceptance criteria and corrective action procedures for out-of-control surrogate spike results are listed in Table 8-2.

Frequencies for surrogate spike analyses will be consistent with the referenced method protocols.

8.4.5 Internal Standards

Internal Standards (ISs) are compounds of known concentrations used to quantitate the concentrations of target detections in field and QC samples. ISs are added to all samples after sample extraction or preparation. Because of this, ISs provide for the accurate quantitation of target detections by allowing for the effects of sample loss through extraction, purging, and/or matrix effects. ISs are used for any method requiring an IS calibration. Corrective action is required when ISs are out of control. Acceptance criteria and corrective action procedures for out-of-control internal standard spike results are listed in Table 8-2.

8.4.6 Instrument Calibration and Frequency

Analytical instruments will be calibrated in accordance with the referenced analytical methods. All target analytes that are reported to EPA will be present in the initial and continuing calibrations, and these calibrations must meet the acceptance criteria specified in referenced methods. Records of standard preparation and instrument calibration will be maintained by the contract laboratory. Records will unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards will be traceable to standard materials.

Analyte concentrations are determined with either calibration curves (linear regression) or response factors (RFs). All correlation coefficients for linear regression calibration curves or relative standard deviation (RSD) of RFs to determine linearity must meet the acceptability criteria specified within the method. For GC/MS methods, the average RF from the initial five-point calibration will be used to determine analyte concentrations. The continuing calibration curve will not be used to update the RFs from the initial five-point calibration. GC/MS methods also will meet all instrument performance and/or tuning criteria as specified by the methods.

Initial Calibration Verification

Initial calibration curves must be verified using a standard made from a source independent of the one used to make the initial calibration standards. All target compounds must be included within the initial calibration verification (ICV), typically at a concentration around the midpoint of the calibration curve. Control limits and corrective action procedures for out-of-control initial calibration verification results are listed in Table 8-2.

Continuing Calibration and Verification

Initial calibration curves must be verified daily prior to sample analysis. All target compounds must be included, typically at a concentration around the midpoint of the calibration curve. Continuing calibration verifications (CCVs) are check samples required at frequencies specified in each analytical method, typically at the beginning and end of each analytical sequence and

after every ten samples analyzed (as specified in each analytical method). Control limits and corrective action procedures for out-of-control CCV results are listed Table 8-2.

Calibration procedures for a specific laboratory instrument will consist of initial calibration (3- or 5-points), initial calibration verification (ICV) and continuing calibration verification (CCV). Calibration protocols included in method references, including calibration frequencies, conditions, and acceptance criteria, will be followed.

8.5 Quality Assurance Objectives For Measurement Data

This section identifies specific objectives for precision, accuracy, representativeness, completeness, and comparability of measurement data collected to support the Phase I data quality objectives.

8.5.1 Precision

Precision is defined as the agreement between a set of replicate measurements without assumption or knowledge of the true value. Agreement is expressed as either the relative percent difference (RPD) for duplicate measurements, or the range and standard deviation for larger numbers of replicates. Precision will be assessed through the calculation of the relative percent difference (RPD) for two replicate samples. RPD is calculated according to the following formula:

$$RPD = \frac{(S - D)}{(S + D)/2} \cdot 100$$

where: S = Original sample value
D = Duplicate sample value

Field precision is assessed through the collection and measurement of field duplicates. The variability between field duplicates reflect the combined variation in concentration between nearby samples and the variation due to measurement error. Because the variability between field duplicates is random and may be either small or large, no quantitative requirement for the agreement of field duplicates is established for this project.

Precision in the laboratory is assessed through calculation of RPDs for duplicate analyses or relative standard deviations (RSDs) for three or more replicate analyses of the same sample. Results from mine waste, soil, and sediment duplicate samples are expected to be more variable than results from duplicate water samples due to the physical and chemical heterogeneity of the solid matrices. Based on this, an RPDs of 50% for mine waste, soil, sediment field duplicate samples and RPDs of 25% for water field duplicates will be used as advisory limits for analytes

detected in both the original sample and its field duplicate at concentrations greater than 5 times the reported quantitation limit.

Differences greater than these advisory limits will be noted for data users through the data validation process.

8.5.2 Accuracy

Accuracy is a measure of the agreement between a measurement and the “true” value. The accuracy of a measurement may be affected by errors introduced by field contamination, sample preparation and handling, and sample analysis. The accuracy of an analytical method is generally assessed by analyses of samples with known concentration levels, including field calibration standards (for field based measurements), laboratory control samples, MS/MSD samples, and PE samples.

The accuracy required for data usability depends on a number of factors. In general, good accuracy is most important for samples whose concentration values are close to the level of concern, and a somewhat lesser level of accuracy may be acceptable for samples whose concentrations are either well below or well above a level of concern. Based on this, the goal of Phase I is to achieve an analytical accuracy of $\pm 25\%$ for analytes that are within a factor of 10 of initial estimates of the level of concern, and $\pm 50\%$ for samples either 10-fold above or 10-fold below initial estimates of the level of concern.

8.5.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent characteristics of a population, parameter variations at a sampling point, or an environmental condition. Representativeness of field measurements is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the SAP and SOPs are followed. The Phase I sampling activities are designed to provide data that are representative of conditions at specific locations and times of sample collection.

8.5.4 Completeness

Data are considered complete when a prescribed percentage of the total intended measurements and samples are obtained. Analytical completeness is defined as the percentage of valid analytical results requested.

Field completeness is a measure of the amount of valid measurement data collected for the project. The target completeness objective for field measurements collected for this sampling program is 95 percent or more.

Laboratory completeness is a measure of the amount of valid laboratory-measurement data obtained for the project. For this sampling program, a minimum of 90% percent of the planned collection of individual samples for quantification must be obtained to achieve a satisfactory level of data completeness.

8.5.5 Comparability

Data are comparable if collection techniques, measurement procedures, methods, and reporting units are equivalent for the samples within a sample set. These criteria allow comparison of data from different sources. Comparable data will be obtained by specifying standard units for physical measurements and standard procedures for sample collection, processing, and analysis.

The criteria for field comparability will be to ensure and document that the sampling designs are properly implemented and the sampling procedures are consistently followed for the duration of the Phase I data collection program. Each sampling task will utilize standardized procedures for sample collection and field measurements, as specified in Section 5 of this plan.

The criteria for laboratory data comparability will be to ensure that the laboratory results generated during Phase I will be comparable to laboratory data collected for future environmental investigations at OU3 and comparable to the asbestos data already collected by EPA in the vicinity of OU3. This goal will be achieved through utilization of standard EPA Test Methods and site-specific asbestos analysis methods for sample analyses and adherence to quality assurance/quality control and analytical procedures specified for the OU3 RI.

9.0 DATA MANAGEMENT

9.1 Data Applications

All data generated as part of the Phase IIA sampling event will be maintained in an OU3-specific Microsoft® Access database. This will be a relational database with tables designed to store information on station location, sample collection details, preparation and analysis details, and analytical results. Results will include asbestos data (including detailed structure attributes for TEM analyses and optical properties for PLM analyses) and non-asbestos chemical data (e.g., metals).

As needed, EPA staff and designated contractors will develop tabular and graphical data summaries, perform statistical analyses, and generate maps using commercially-available applications such as Microsoft® Access and Excel and ArcGIS®.

9.2 Roles and Responsibilities for Data Flow

9.2.1 Field Personnel

W.R. Grace contractors will perform all Phase IIA sample collection in accordance with the project-specific sampling plan and SOPs presented above. In the field, sample details will be documented on hard copy media-specific FSDS forms and in field log books (see Section 5.5). COC information will be documented on hard copy forms (see Section 5.6). FSDS and COC information will be manually entered into a field-specific³ OU3 database using electronic data entry forms. Use of electronic data entry forms ensures the accuracy of data entry and helps maintain data integrity. For example, data entry forms utilize drop-down menus and check boxes whenever possible. These features allow the data entry personnel to select from a set of standard inputs, thereby preventing duplication and transcription errors and limiting the number of available selections (e.g., media types). In addition, entry into a database allows for the incorporation of data entry checks. For example, the database will allow a unique sample ID to only be entered once, thus ensuring that duplicate records cannot be created.

Entry of FSDS forms and COC information will be completed weekly, or more frequently as conditions permit. Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site weekly. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, COC forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS_20070831.pdf). Electronic

³ The field-specific OU3 database would be a simplified version of the master OU3 database. This simplified database will include only the station and sample recording and tracking tables, as well as the FSDS and COC data entry forms.

copies of all digital photographs will also be posted weekly to the project-specific FTP site. File names for digital photographs will include the station identifier, the sample date, and photograph identifier (e.g., ST-1_20070831_12459.tif).

After FSDS data entry is completed, a copy of the field-specific OU3 database will be posted to the project-specific FTP weekly, or more frequently as conditions permit. The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB_20070831.mdb).

9.2.2 Laboratory Personnel

Each of the laboratories performing asbestos analyses for the Phase IIA sampling event are required to utilize all applicable Libby-specific Microsoft® Excel spreadsheets for asbestos data recording and electronic submittals (see Section 6.7). Upon completion of the appropriate analyses, EDDs will be transmitted via email to a designated email distribution list within the appropriate turn around time. Hard copies of all analytical laboratory data packages will be scanned and posted as a PDF to the project-specific FTP site. File names for scanned analytical laboratory data packages will include the laboratory name and the job number to facilitate document organization (e.g., LabX_12365-A.pdf).

9.2.3 Database Administrators

Day-to-day operations of the master OU3 database will be under the control of EPA contractors. The primary database administrator will be responsible for sample tracking, uploading new data, performing error checks, and making any necessary data corrections. New records will be added to the master OU3 database within an appropriate time period of FSDS and/or EDD receipt.

Incremental backups of the master OU3 database will be performed daily Monday through Thursday, and a full backup will be performed each Friday. The full backup tapes will be stored off-site for 30 days. After 30 days, the tape will be placed back into the tape library to be overwritten by another full backup.

Each Friday, a copy of the master OU3 database will be posted to a project-specific FTP site to allow timely access to results by data users. The master OU3 database posted to the FTP site will include the post date in the file name (e.g., MasterOU3DB_20070831.mdb).

9.3 Data Storage

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the Project Manager. At the termination of this project, all original data records will be provided to the Project Manager for incorporation into the site project files.

10.0 ASSESSMENT AND OVERSIGHT

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required and that deviations from procedures are documented. These reports also serve to keep management current on field activities. Assessment, oversight reports, and response actions are discussed below.

10.1 Assessments

10.1.1 Field Oversight

All individuals who collect samples during field activities will be provided a copy of this SAP and will be required to participate in a pre-sampling readiness review meeting to ensure that methods and procedures called for in this SAP and associated SOPs are understood and that all necessary equipment is on hand. EPA may perform random and unannounced field audits of field sampling collection activities, as may be deemed necessary.

10.1.2 Laboratory Oversight

All laboratories selected for analysis of samples for asbestos will be part of the Libby analytical team. These laboratories have all demonstrated experience and expertise in analysis of LA in environmental media, and all are part of an on-going site-specific quality assurance program designed to ensure accuracy and consistency between laboratories. These laboratories are audited by EPA and NVLAP on a regular basis. Additional laboratory audits may be conducted upon request from the EPA, as may be needed.

10.2 Response Actions

If any inconsistencies or errors in field or laboratory methods and procedures are identified, response actions will be implemented on a case-by-case basis to correct quality problems. All response actions will be documented in a memo to the EPA RPM for OU3 at the following address:

Bonita Lavelle
U.S. EPA Region 8
1595 Wynkoop Street
Denver, CO 80202-1129
E-mail: lavelle.bonita@epa.gov

Any problems that cannot be corrected quickly through routine procedures may require implementation of a corrective action request (CAR) form.

10.3 Reports to Management

Field and analytical staff will promptly communicate any difficulties or problems in implementation of the SAP to EPA, and may recommend changes as needed. If any revisions to this SAP are needed, the EPA RPM will approve these revisions before implementation by field or analytical staff.

11.0 DATA VALIDATION AND USABILITY

11.1 Data Validation and Verification Requirements

Data validation, review, and verifications must be performed on sample results before distribution to the public for review.

Validation of Non-Asbestos Data

For non-asbestos analytical data, data validation will be performed in accord with the most current versions of EPA's National Functional Guidelines. In brief, the validation process consists of examining the sample data package(s) against pre-determined standardized requirements. The validator may examine, as appropriate, the reported results, QC summaries, case narratives, COC information, raw data, initial and continuing instrument calibration, and other reported information to evaluate the accuracy and completeness of the data package. During this process, the validator will determine if analytical methodologies were followed and QC requirements were met. The validator may recalculate selected analytical results to verify the accuracy of the reported information, as appropriate, and will assign qualifiers to the data as needed.

Verification of Asbestos Data

For asbestos analytical data, data verification includes checking that all required data have been entered on the laboratory bench sheets and field sample data sheets, and that results have been transferred correctly to the EDD. Some of the data verification checks are performed as a function of built-in quality control checks in the Libby-specific data entry spreadsheets. Additional verifications of field and analytical results will be performed manually by independent review of the bench sheets and FSDS. The initial frequency of manual review will be 10% of all samples. This initial rate may be revised either upward or downward depending on the frequency and nature of errors that are identified by the verification process.

11.2 Reconciliation with Data Quality Objectives

Once all samples have been collected and the analytical data have been reported and validated, the data will be reviewed by data users to determine if DQOs were achieved. [

Note to EPA--the Phase I SAP refers to a Phase I data report (in yellow, below). If we are not going to have a stand alone Phase I data report, will the data summary in Phase II include a full evaluation of data quality results?]

The Phase I data summary report will include a qualitative and quantitative review of all QC samples and all deviations from sampling and analysis plans

described in this report, along with conclusions regarding the reliability of the data for their intended use.

12.0 REFERENCES

- Asbestos Hazardous Emergency Response Act (AHERA). 1986. Title 20, Chapter 52, Sec. 4011. Public Law 99-519.
- Amandus HE, Wheeler R. 1987. The Morbidity and Mortality of Vermiculite Miners and Millers Exposed to Tremolite-Actinolite: Part II. Mortality. *Am. J. Ind. Med.* 11:15-26.
- Camp Dresser & McKee, Inc. (CDM). 2002. Final Sampling and Analysis Plan for the Remedial Investigation of Contaminant Screening Study (CSS), Libby Asbestos Site, Operable Unit 4. April 2002.
- Camp Dresser & McKee, Inc. (CDM). 2003. Final Sampling and Analysis Plan, Remedial Investigation, Libby Asbestos Site OU4. May 2003.
- Camp Dresser & McKee, Inc. (CDM). 2007. Site-Wide Quality Assurance Project Plan, Libby, Montana Asbestos Project. *Final Report Date Pending TAU Review*.
- International Organization for Standardization. 1995. Ambient Air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).
- McDonald JC, Harris J, Armstrong B. 2004. Mortality in a cohort of vermiculite miners exposed to fibrous Amphibole in Libby, Montana. *Occup. Environ. Med.* 61:363-366.
- McDonald JC, McDonald AD, Armstrong B, Sebastien P. 1986. Cohort study of mortality of vermiculite miners exposed to tremolite. *Brit. J. Ind. Med.* 43:436-444.
- Montana Department of Environmental Quality (MDEQ). 2003. Montana Tier 1 Risk-Based Corrective Action Guidance for Petroleum Releases, October 2003.
<http://www.deq.state.mt.us/rem/hwc/rbca/NewRBCA11-2003/Tier1Guidance10-03.pdf>
- Meeker GP, Bern AM, Brownfield IK, Lowers HA, Sutley SJ, Hoeffen TM, Vance JS. 2003. The Composition and Morphology of Amphiboles from the Rainy Creek Complex, Near Libby, Montana. *American Mineralogist* 88:1955-1969.
- Peipins LA, Lewin M, Campolucci S, Lybarger JA, Miller A, Middleton D, et al. 2003. Radiographic abnormalities and exposure to asbestos-contaminated vermiculite in the community of Libby, Montana, USA. *Environ. Health Perspect.* 111:1753-1759.
- Quivik L.F., 2002. Expert Report. United States v. W.R. Grace Civil Action No.90-11-2-07106/2. July 29, 2002.

Rohs AM, Lockey JE, Dunning KK, Shulka R, Fan H, Hilbert T, Borton E, Wiot J, Meyer C, Shipley RT, LeMasters GK, Kapol V. 2007. Low level Fiber Induced Radiographic Changes Caused by Libby Vermiculite: A 25 year Follow-up Study. Am J Respiratory and Critical Care Medicine. Published online December 6, 2007 as doi:10.1164/rccm.200706-814OC.

Schafer and Associates, 1992a. Engineering Analysis of Flood Routing Alternatives for the W.R. Grace Vermiculite Tailings Impoundment. March 1992.

Shafer and Associates, 1992b. W.R. Grace Vermiculite Mine Closure Water Quality Data Report No. 1. November 1991. March 4, 1992.

Shafer and Associates, 1992c. W.R. Grace Vermiculite Mine Closure Water Quality Data Report No. 2. March 1991. May 6, 1992.

Shafer and Associates, 1992d. W.R. Grace Vermiculite Mine Closure Water Quality Data Report No. 3. July 1992. November 6, 1992.

Shafer and Associates, 1993. W.R. Grace Vermiculite Mine Closure Water Quality Data Report No. 4. October 1992. January 13, 1993.

Shafer and Associates, 1994. W.R. Grace Vermiculite Mine Closure Water Quality Data Report No. 5. October 1993. January 27, 1994.

Shafer and Associates, 1995. W.R. Grace Vermiculite Mine Closure Water Quality Data Report No. 6. September 1994. February 3, 1995.

Sullivan PA. 2007. Vermiculite, Respiratory Disease and Asbestos Exposure in Libby, Montana: Update of a Cohort Mortality Study. Environmental Health Perspectives doi:10.1289/ehp.9481 available online at <http://dx.doi.org>.

U.S. Environmental Protection Agency (EPA). 1986. Test Methods for Evaluating Solid Waste Physical/Chemical Methods and Updates I, II, IIA, IIB, III, IIIA and IIIB, EPA SW-846. <http://www.epa.gov/epaoswer/hazwaste/test/main.htm>

U.S. Environmental Protection Agency (EPA). 1994a. Method 100.2: Determination of Asbestos Structures Over 10 um in Length in Drinking Water. U.S. Environmental Protection Agency, Office of Research and Development. June 1994.

U.S. Environmental Protection Agency (EPA). 1994b. Methods for the Determination of Metals in Environmental Samples, Supplement 1. EPA/600/R-94/111. May 1994.

U.S. Environmental Protection Agency (EPA). 2000a. Sampling and Quality Assurance Project Plan, Revision 1 for Libby, Montana, Environmental Monitoring for Asbestos, Baseline Monitoring for Source Area and Residential Exposure to Tremolite-Actinolite Asbestos fibers. January 2000.

U.S. Environmental Protection Agency (EPA). 2000b. Methods for Measuring Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates – Second Edition. U.S. Environmental Protection Agency, Office of Research and Development. EPA 600/R-99/064. March 2000.

U.S. Environmental Protection Agency (EPA). 2001. EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-01/003. March 2001.

U.S. Environmental Protection Agency (EPA). 2005. Supplemental Remedial Investigation Quality Assurance Project Plan (SQAPP) for Libby, Montana. June 24, 2005.

U.S. Environmental Protection Agency (EPA). 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-06/001. February 2006.

U.S. Environmental Protection Agency (EPA). 2007. Sediment Toxicity Identification Evaluation (TIE) – Phases I, II, and III Guidance Document. U.S. Environmental Protection Agency, National Health and Environmental Effects Research laboratory (NHEERL). EPA/600/R-xx/xxx. Draft – June 2007.

U.S. Geological Survey (USGS). 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, U.S. Geological Survey, Department of the Interior, Reston, VA.

Ward TJ, T Spear, J Hart, C Noonan, A Holian, M Getman, and JS Webber. 2006. Trees as reservoirs for amphibole fibers in Libby, Montana. Science of the Total Environment 367: 460–465.

W.R. Grace, 2006. Letter from Robert R. Marriam, Remedium, to Bonnie Lavelle, EPA dated August 2006.

Zinner E,R. 1982. Geohydrology of the Rainy Creek Igneous Complex Near Libby Montana. Masters Thesis Univeristy of Reno, Nevada. June 1982.

Zucker, G. 2006. Expert Report Prepared for W.R. Grace Company. Provided by Robert Marriam, Remedium correspondence to Bonita Lavelle, EPA, October 24, 2006.

ATTACHMENT A

STANDARD OPERATING PROCEDURES

SOP Description	SOP ID
Soil Sampling for Analyses of Non-Volatile Constituents	No. 1 (Rev. 0)
Soil Sampling for Volatile Organic Compounds	No. 2 (Rev. 0)
Surface Water Sampling	No. 3 (Rev. 0)
Surface Water Discharge Measurement	No. 4 (Rev. 0)
Sediment Sampling for Chemical Analysis	No. 5 (Rev. 0)
Groundwater Sampling for Chemical Analysis	No. 6 (Rev. 0)
Equipment Decontamination	No. 7 (Rev. 0)
Sample Handling and Shipping	No. 8 (Rev. 0)
Field Documentation	No. 9 (Rev. 1)
Field Equipment Calibration	No. 10 (Rev. 0)
GPS Data Collection	No. 11 (Rev. 0)
Investigation Derived Waste (IDW) Management	No. 12 (Rev. 0)
Groundwater Elevation Measurements	No. 13 (Rev. 0)
Collection of Outdoor Ambient Air Samples	AMB-LIBBY-OU3 (Rev. 0)
Sampling, Preparation, and Analysis of Tree Bark for Asbestos	TREE-LIBBY-OU3 (Rev. 0)
Preparation and Analysis of Organic Debris for Asbestos	DEBRIS-LIBBY-OU3 (Rev. 0)
Soil Sample Preparation	ISSI-LIBBY-01 (Rev. 8)
Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy (PLM)	SRC-LIBBY-01 (Rev. 2)
Analysis of Asbestos Fibers in Soil By Polarized Light Microscopy (PLM)	SRC-LIBBY-03 (Rev. 2)
SOP for Water Toxicity Testing	TBD